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(Patent)

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Patent Application of:
Manabu MATSUI et al.

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For:	HEAT FUSIBLE CONJUGATE FIBER	Examiner:	J.STEELE

DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Keisuke Nagashima, hereby declare as follows:

I graduated from graduate school of engineering of Gunma University and obtained a master of engineering degree in March 2005.

I have been engaged in researching and developing fibers and nonwoven fabrics using the same as a researcher of Kao Corporation from April 2005 to the present.

The following experiment was carried out by me or under my direct supervision. Test procedures and results are shown below.

The sample for the measurement of heat shrinkage was the same fiber as Example 5 of the present specification. The measurement of heat shrinkage was conducted in accordance with JIS L 1015. An English version of JIS L 1015 is enclosed.

JIS L 1015 stipulates two ways of measurement: (1) shrinkage percentage by hot water and (2) shrinkage percentage at hot dry (pages 26-27 of the English version of JIS L 1015). Kajita does not explicitly indicate whether measurement (1) or (2) was employed. As such, the measurement was conducted by the method of measurement (1), which is the ordinary method. Further, JIS L 1015 stipulates the temperature of hot water as a "suitable temperature" without indicating a specific temperature. Kajita also does not indicate a specific temperature. In

general, the method of shrinkage percentage by hot water is also called "shrinkage percentage by boiling water," and there are many cases in the field of textile material in which the measurement is conducted at a temperature around 100°C. In view of this, this experiment was conducted at a temperature of 98°C. After the fiber was taken out of hot water, a small amount of water that was attached to the fiber was removed by absorbent paper. Then, the fiber was dried over 48 hours in the atmosphere under the condition of 22°C/65%/RH. The fiber length before and after heating by hot water was measured using a thermomechanical analysis (TMA) apparatus. The fiber was cramped by this apparatus under a load of 0.14 mN/tex. The shrinkage percentage by hot water was calculated by (L-L')/L*100. The definition of L and L' is provided on page 26 of the English version of JIS L 1015. The result of the measurement is shown in the table below.

	L (mm)	L' (mm)	shrinkage percentage (%)
The Claimed Fiber	24.999	25.754	-3.0

As is apparent from the above result, the claimed fiber showed a negative value of heat shrinkage measured by the same method as Kajita. The negative value of heat shrinkage means that the fiber increases in length upon being subjected to heat. This result clearly shows that Kajita does not inherently involve the claimed fiber.

It is considered that the major reason of the difference in heat shrinkage between the claimed fiber and Kajita is due to the difference of the second resin. In the claimed fiber, the second resin is polypropylene but not ethylene-propylene random copolymer that is used in Kajita. The behavior of heat shrinkage is mainly ruled by the second resin rather than the first resin. The same reason will be applied to the secondary reference, "Polypropylene An A-Z Reference." The secondary reference only discloses the relationship between the spinning speed and the orientation of polypropylene itself. It is not reasonable to apply the data of polypropylene itself to a conjugated fiber since shrinkage behavior of the conjugated fiber depends on the manner of consolidation in the course of melt spinning, in addition to temperature and drawing speed of melt spinning. The manner of consolidation is influenced by the kind of resins contained in the conjugated fiber. In the claimed invention, the second resin is polyethylene but not polypropylene. The second resin of polyethylene has a controlled lower value of the orientation index. The controlled lower value of the orientation index enables the desired manner of consolidation of the conjugated fiber in the course of melt-spinning. A suitable control of the orientation index successfully results in producing a heat-extensible fiber.

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S. Code 1001 and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

By: Keisuke Nagashima
Keisuke Nagashima

Date: February 15, 2011

Enclosure: English version of JIS L 1015

UDC 677-1:62-486.1:677.4:620.1

JIS

This standard was revised in 3, 1992
and once again in Apr., 1999

JAPANESE INDUSTRIAL STANDARD

Testing Methods for Man-Made Staple Fibres

JIS L 1015-1981

Translated

by

Japanese Standards Association

1-24, Akasaka 4, Minato-ku
Tokyo 107 Japan

This English translation is prepared at the expense of Japan External Trade Organization (JETRO).

UDC 677-1:62-486.1:677.4:620.1

JAPANESE INDUSTRIAL STANDARD | J I S

Testing Methods for Man-Made Staple Fibers | L 1015-1981

1. Scope

This Japanese Industrial Standard specifies the testing methods for man-made staple fibers.

Remarks 1. This is also applicable to a single fiber in the tow of man-made fibers.

2. In this standard the units and numerical values in { } are in accordance with the International System of Units (SI), and are given for reference only.

2. Definition

The definitions of the terms used in this standard shall be as follows:

(1) standard condition of laboratory This shall be the condition under standard temperature and humidity of grade 2 (temperature 20 ± 2°C, relative humidity 65 ± 2 %) specified in JIS Z 8703.

(2) standard condition of sample Carrying out preliminary drying in a dryer at 40 ± 5°C, lower its water content down to the official regain or less, place the sample in a laboratory or apparatus under the standard condition and finally obtain moisture equilibrium.

Remarks 1. In the cases of viscose rayon, cupro, and acetate, the temperature for drying shall be 65 ± 5°C.

2. In the case of the fibers whose official regain is zero, no predrying is needed.

(3) moisture equilibrium The condition of the sample when, after standing in the laboratory kept under a difinite condition, the mass of the sample is weighed every hour or less often, and the difference of masses weighed before and after the interval has become 0.1 % or under of the mass weighed after.

(4) absolute dry condition of the sample This is the condition where constant weight is obtained after standing in a hot-air dryer at 105 ± 2°C.

Remark: In the case of the fiber whose official regain is zero, the standard condition is regarded as the absolute dry condition.

(5) absolute dry mass This is the mass at the absolute dry condition.

Applicable Standards: See pages 55 to 57.

(6) constant weight condition The condition where after heating in a dryer, or others under a definite condition, the difference of masses of the sample weighed every 15 minutes or less often has become 0.1 % or under of the mass weighed after the interval.

(7) constant weight This is the mass at a constant weight condition.

(8) official regain This is the moisture of a fiber decided officially.

(9) corrected weight This is be the weight of absolute dry mass plus mass corresponding to the official regain.

(10) fineness This is the unit representing the thickness of a fiber. The fineness used in this standard shall be as follows:

(a) denier This is the number of grams per 9000 m in length.

(b) tex This is the number of grams per 1000 m in length. One denier is equivalent to 0.11 tex.

(11) apparent fineness This is the fineness at an arbitrary condition.

(12) fineness based on corrected weight This is the fineness under the condition of the official regain.

(13) pre-tension This is the load that makes a fiber not elongate but straighten.

3. Marking of Fineness

Fineness shall be marked as follows:

(1) In the Case of Using Denier 1.5 denier 1.5 D

(2) In the Case of Using Tex Follow the description in 7. Method of Marking in JIS L 0101.

4. Preparation of Sample

Prior to the tests(1) easily influenced by temperature or humidity, place the sample under standard condition prior to testing. However, in case of the tests such as moisture ragain, the percentage of free water, and the moisture regain in bale, quickly pick suitable quantity from an original sample and keep them in a tightly stoppered container.

Note (1) The tests easily influenced by temperature and moisture shall be as follows: fiber length, fineness, fineness of two corrected weight, tensile strength and elongation percentage, knot strength, loop strength, modules of elongation elasticity, initial tensile resistivity, crimp, coefficient of friction, tensile strength at wet condition after 5 % alkali treatment, 5 % elongation stress at wet condition, degree of water swelling, degree of alkali swelling, and so on.

Remark: When the laboratory or apparatus under standard condition is not available, let the sample become constant temperature (20 $\pm$ 2°C) by keeping in a closed container (placing 36 % sulfuric acid in it). In this case, this pursuit shall be noted supplementarily.

5. Testing Condition

5.1 Testing Place The tests(1) influenced by temperature and humidity shall be carried out in the laboratory kept at standard condition.

Remark: When the tests(1) influenced by temperature and humidity are carried out in the place other than the laboratory kept at standard condition, the temperature and humidity for testing shall be appended with a note.

5.2 Absolute Dry Mass For obtaining absolute dry mass, not only a hot air dryer but also infrared dryer, high-frequency dryer, or reduced pressure dryer may be used. In this case, the condition shall be added in a note.

In the case of the fiber that is sensitive to temperature, make use of the temperature lower than 105°C, and append the condition with a note.

5.3 Official Regain The official regain to in relation to the absolute dry mass shall be as shown in Table 1.

Table 1

Kind of fibers	Official regain %
Rayon	11.0
(Polynosic)	11.0
Cupro	11.0
Acetate	6.5
Triacetate	3.5
Nylon	4.5
Vinylon	5.0
Vinylidene	0
Polyvinyl chloride	0
Polyester	0.4
Acrylic	2.0
Modacrylic	2.0
Polypropylene	0
Polychlal	3.0

5.4 Initial Tension The following loads shall be adopted as the initial tension.

Rayon, cupro, and acetate

at standard condition ..... Gram number of 1/20 of nominal denier number { 4.41 mN of nominal tex number }

at wet condition .... Gram number of 1/40 of nominal denier number { 2.21 mN of nominal tex number }

Nylon, vynylon, vinylidene, and polychlal

at both standard and wet condition .. Gram number of 1/20 of nominal denier number { 4.41 mN of nominal tex number }

Polyvinyl chloride, polyester, polypropylene

at both standard and wet condition .. Gram number of 1/15 of nominal denier number { 5.88 mN of nominal number }

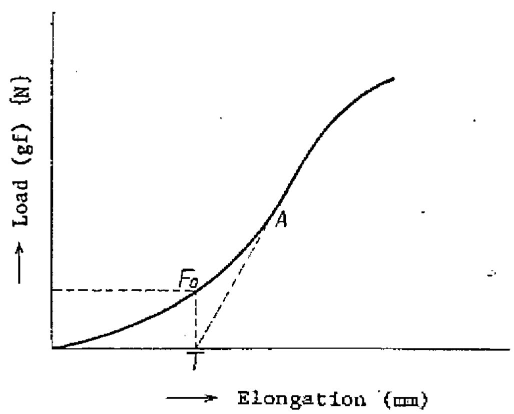
Acrylic, modacrylic

at both standard and wet condition ..	Gram number of 1/10 of nominal denier number 8.82 mN of nominal number

In case, however, of unsuitableness of this load, the following shall be adopted; plotting the curve of load and elongation at initial stage as shown in Fig. 1, draw a tangent at the point *A* (the maximum tangential angle point) where the changing ratio of a load to an elongation is maximum, and find the point *T* where the tangent intersects the elongation axis. Draw a perpendicular from *T*, and, after finding the point F_0 where the perpendicular intersects the curve, make use of the load corresponding to F_0' as the initial tension.

Remark: This is applicable to the tests such as fineness of tow corrected weight, tensile strength and elongation percentage, knot strength, loop strength, modulus of elongation elasticity, initial tensile resistivity, coefficient of friction, tensile strength at wet condition after 5 % alkali treatment, and others.

Fig. 1



5.5 Measurement of Temperature and Humidity Measure the temperature and humidity by using a Meteorological Angency type prescribed in JIS Z 8806 or an Assmann type psychrometer, and find relative humidity from the humidity table by Sprung' formula.

6. Testing Items

Testing items shall be as follows:

(1) Moisture regain and percentage of free water (applicable to synthetic fibers)

(2) Moisture regain in bale

(3) Equilibrium moisture regain

(4) Fiber length

(5) Fineness

(6) Fineness of tow corrected weight

(7) Tensile strength and elongation percentage

(8) Knot strength

(9) Loop strength

(10) Modulus of elongation elasticity

(11) Initial tensile resistivity

(12) Crimp

(13) Coefficient of friction

(14) Specific gravity and density

(15) Shrinkage percentage

(16) Melting point and thermal shrinkage temperature (applicable to synthetic fibers)

(17) Whiteness

(18) Glossiness

(19) Abnormal fibers

(20) Ash content

(21) Titanium oxide

(22) Solvent extraction content

(23) Tensile strength at wet condition after 5 % alkali treatment (applicable to rayon and cupro)

(24) 5 % elongation stress at wet condition (applicable to rayon and cupro)

(25) Degree of water swelling (applicable to rayon and cupro)

(26) Degree of alkali swelling (applicable to rayon and cupro)

(27) Skin ratio (applicable to rayon)

(28) Sulfur content (applicable to rayon)

(29) Mean polymerization degree (applicable to rayon and cupro)

(30) Degree of exhaustion (applicable to rayon and cupro)

(31) Degree of acetylation (applicable to acetate)

7. Testing Methods

7.1 Moisture Regain and Percentage of Free Water

7.1.1 Moisture Regain Take about 5 g of the sample, and after weighing its mass and absolute dry mass, calculate the moisture regain (%) according to the following formula. Carry out twice the test and express by the average of them (to the first decimal place).

$$\text{Moisture regain (\%)} = \frac{W-W'}{W'} \times 100$$

where W: mass of sample when sampled (g)

W': absolute dry mass of sample (g)

Remark: This is applicable to the synthetic fibers other than those of 0 % of official regain.

7.1.2 Percentage of Free Water Take about 5 g of the sample and, after weighing its mass and the mass at standard condition, calculate free water percentage (%) according to the following formula. Carry out twice the test, and express the average of them (to the first decimal place).

$$\text{Percentage of free water (\%)} = \frac{W-W'}{W'} \times 100$$

where W: mass of sample when sampled (g)

W': mass of sample under standard condition (g)

Remark: This is applicable to the synthetic fibers of 0 % official regain.

7.2 Moisture Regain in Bale

Divide laterally the content of a sample bale half of which has been opened, and pick up quickly four samples of 50 to 100 g respectively as shown in Fig. 2 and, after weighing its mass and absolute dry mass, calculate the moisture regain in bale (%) according to following formula. Express the average of four measured values (to the first decimal place).

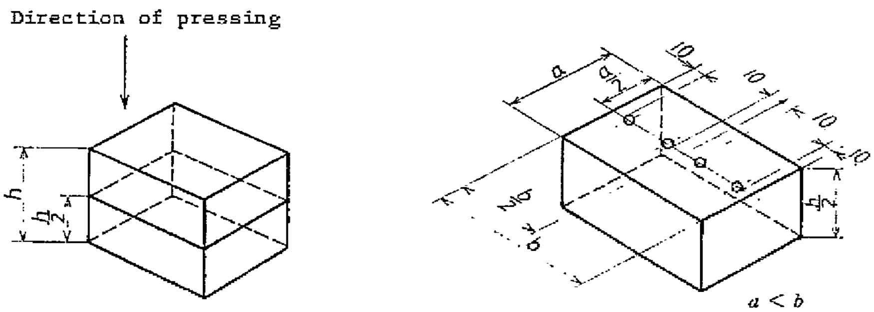
$$\text{Moisture regain in bale (\%)} = \frac{W - W'}{W'} \times 100$$

where W : mass of sample when sampled (g)

W': absolute dry mass of sample (g)

Fig. 2

Unit: mm



7.3 Equilibrium Moisture Regain Take about 5 g of the sample that has been under moisture equilibrium and, after weighing its mass and absolute dry mass, calculate the equilibrium moisture regain (%) according to the following formula. Carry out twice the test, and express the average of them (to the first decimal place).

$$\text{Equilibrium moisture regain (\%)} = \frac{W - W'}{W'} \times 100$$

where W : mass of sample when sampled (g)

W': absolute dry mass of sample (g)

7.4 fiber Length

7.4.1 Mean Fiber Length

Method A (Staple Diagram Method) Card the definite quantity(2) of the sample, which is accurately weighed, by using a metal comb, and prepare a staple diagram of 25 cm wide by a Bear-type sorter. For preparing, the frequency of drawing with a clip in order to arrange all fibers on a velvet plate is about 70 times. On the plate, put the celluloid plate graduated by scale, and draw a diagram on a graph paper.

Divide equally the staple diagram made by above method into 50 divisions according to the length of each fiber, and measure the length of fibers that are positioned at both ends of this diagram and on the border of two adjacent divisions. Calculate the mean fiber length (mm) after dividing by 50 the total of the average of two fibers at both ends plus summed length of 49 fibers at borders of divisions. Carry out twice the test, and express the average of them (to the first decimal place).

Note (2) For rayon, cupro, and acetate, use the quantity obtained from the nomograph on Attached Figure, and for other fibers use the quantity which is obtained after multiplying the value of nomograph by $\rho/1.5$ [ρ : density of fiber (g/cm^3)] and which is rounded to 10 mg unit according to JIS Z 8401.

<u>Method B (Corrected Staple Diagram Method)</u> After having obtained the mean fiber length according to method A, pull out one fiber each from ten points on the straight part of the diagram, and measure the length of each fiber after making it straight fixing one end with a finger. After obtaining the average of these straightened ten fibers and then the average of them on the diagram, calculate the mean fiber length (mm) according to following formula. Carry out twice the test, and express the average of them (to the first decimal place).

Mean fiber length (mm) = $\frac{\bar{l'}}{\bar{l}} \times L_0$

where $\bar{l}$: average of the lengths of ten fibers pulled out which are measured on digram (mm)

$\bar{l'}$: average of the lengths of ten fibers pulled out (mm)

L_0: mean fiber length by Method A (mm)

Remarks 1. When preparing the diagram referred to, it is admissible to use a thickn ground paper applied vaseline and to arrange fibers on it in avobe-mentioned method to prepare staple diagram.

2. For the fiber having a serious crimp or thick fineness, Method B is suitable.

Method C (Direct Method) Pull out at random a single fiber one by one, and make it straight without elongation, measure the fiber length on a scale to the unit of mm. Meausre 200 fibers, and express the average of them (to the first decimal place).

Remark: For the fiber which it is difficult to prepare a staple diagram for, apply Method C.

7.4.2 Weight Percentage of Staple-over-the-Limited-Cut-Length

Method A (Staple Diagram method) From the staple diagram prepared in 7.4.1, pick up the fibers which are longer than the mean fiber length plus 5.0 mm as an allowance, in the case of nominal fiber length shorter than 50 mm, and plus 10.0 mm as an allowance in the case of nominal fiber length 50 mm or more. Weigh the mass of these fibers, and express the percentage of the mass to whole mass (to the first decimal place).

Method B (Direct Method) From the fiber length diagram obtained in Method C of 7.4.1, pick up all fibers whose length is longer than the mean fiber length plus 5.0 mm as an allowance in the case of nominal fiber length shorter than 50 mm, and plus 10.0 mm as an allowance in the case of 50 mm or more. Then, sum up the lengths of these fibers. Calculate the weight percentage of staple over-the limited-cut-length (%) according to the following formula (to the first decimal place).

$$\text{Weight percentage of staple over-the-limited-cut-length (\%)} = \frac{L'}{L \times 200} \times 100$$

where L' : sum of fiber length of fibers which are longer than length of mean fiber length plus allowance

L : mean fiber length of single fiber

Remark: This test is applicable to the equal cut fiber but not to the variable cut fiber.

7.5 Fineness

7.5.1 Fineness Based on Corrected Weight

Method A Card in parallel the fibers of some quantity by using a metallic comb and, after putting them on flockpaper placed on a cutting board, press them with a gauge plate as they are stretched with a suitable force. Cut them 30 mm long by using a razor edge or the like, and weigh the mass of 300 pieces of fiber (in the case of shorter fiber, prepare 450 pieces of fiber that are cut to 20 mm in length), from which apparent fineness is calculated.
By means of the equilibrium moisture regain measured separately, calculate the fineness based on corrected weight (D) according to the following formula. Carry out five times the test, and expres the average of them (to the second decimal place).

Fineness based on corrected weight $(D)\,\{\text{tex}\} = D' \times \frac{100+R_c}{100+R_e}$

where D': apparent finess (D) {tex}

R_c: official regain (%)

R_e: equilibrium moisture regain (%)

Remarks 1. For the fiber whose official regain is zero, its apparent fineness shall be regarded to be the fineness based on corrected weight.

2. When preparing 300 pieces of the sample, it is preferable to count 30 pieces ten times for making of one set 300 pieces.

Method B (Easy Method) Making 200 pieces of single fiber one set, weigh the mass of them, and calculate the fineness based on corrected weight (D) by using the mean fiber length measured in 7.4.1, according to the following formula. Carry out five times the test, and express the average of them (to the second decimal place).

Fineness based on corrected weight $(D)\,\{\text{tex}\} = 9000 \times \frac{W}{200 \times L} \times \frac{100+R_c}{100+R_e} \left\{1000 \times \frac{W}{200 \times L} \times \frac{100+R_c}{100+R_e}\right\}$

where W: mass of sample (mg)

L: mean fiber length of single fiber (mm)

R_c: official regain (%)

R_e: equilibrium moisture regain (%)

Remarks 1. In the case of the fiber whose official regain is zero, calculate the fineness based on corrected weight according to the following formula:

Fineness based on corrected weight $(D)\,\{\text{tex}\} = 9000 \times \frac{W}{200 \times L} \left\{1000 \times \frac{W}{200 \times L}\right\}$

2. For the fiber to which Method A is hardly applicable, Method B shall be applied. In the case using Method B, that shall be appended in a note.

[Reference Method]

Fineness (Vibration method)

After attaching a spring load(3) to one end of a single fiber of the sample and attaching the other end to the thread holding clip of a vibration fineness tester, let it go through a vibrator edge and a lower edge, making the vibration length 2.5 cm or 5.0 cm. After letting a spring load suspend to the sample, adjust frequency by turn the frequency dial of a variable low frequency oscillator, and read the resonance frequency by observing the resonance point of the sample on the brown tube. Calculate the apparent fineness (D) according to the following formula 9to the second decimal place).

$$\text{Apparent fineness (D)} \{\text{tex}\} = \frac{M \times 980 \times 9 \times 10^5}{4 l^2 f^2} \left\{ \frac{M \times 980 \times 10^5}{4 l^2 f^2} \right\}$$

where M : mass of spring load (g)

l : length of sample (cm)

f : frequency of oscillator (Hz)

Note (3) The spring load should give no transformation on the sample and be strong enough to eliminate its looseness, and be decided suitably for each denier. Report shall be appended with measuring condition. Generally 0.1 gf/D {8.82 mN/tex }is suitable.

Remarks 1. Obtain the coefficient of variation from the values which are measured from the sample of 50 to 100 pieces, and append the number of pieces measured.

2. In the case of necessity of correction on a bending stiffness, sectional shape and others, calculate a correction factor K by the following formula, and correct by multiplying the apparent fineness obtained from the abovementioned method by this factor.

$$K = \frac{m_0}{m_v}$$

where K : correction factor

m_0 : apparent fineness obtained from mass method

m_v : apparent fineness obtained from vibration method

7.5.2 Coefficient of Variation of Fineness Simultaneously with sampling for measuring fineness, pick up respectively several fibers from 20 spots or more, and bundle them to make a fiber bundle after combing. Pass this in a suitable small hole in a metal plate, and cut perpendicularly both surfaces with a razor edge. By means of a microscope, measure sectional acreas of 50 fibers displayed by Abbe's displaying apparatus([4]), the sectional photograph([4]), or the projection by a profile projector([4]), and calculate a coefficeint of variation (%) (to the first desimal place).

Note ([4]) Make the sectional area of fibers not less than 100 mm^2.

Remark 1. In the case of the fiber whose cross section is a circle, measure the diameter, and calcualte the coefficient of variation (%) from the value squared respectively.

2. Instead of directly measuring area, the following is passable: after cutting out the section copied on a printing paper or graph paper, measure the mass of each section, and calculate the coefficient of variation.

7.6 Fineness of Tow Corrected Weight Take one piece of tow sample, and cut it exactly to 90 cm long under the condition of loading with pre-tension. Calculate apparent fineness from its mass. Then weigh the absolute dry mass of the sample, and calculate the fineness of tow corrected weight (D) according to the following formula. Carry out ten times the test, and obtain the average of them (to the third decimal place).

$$\text{Fineness of tow corrected weight (D)}\ \{\text{tex}\} = W\times 10000\times\frac{100+R_c}{100}\ \left\{W\times 10000\times\frac{100+R_c}{100}\times 0.11\right\}$$

where W: absolute dry mass (g)

R_c: official regain (%)

Remark: In the case of seeking coefficient of variation on tow fineness, prepare 20 pieces of the sample.

7.7 Tensile Strength and Elongation Percentage

7.7.1 Test at Standard Time Draw sectioning lines on glossy and smooth paper as shown in Fig. 3 (spacing distance shall be 20 mm, but if the shortness of the fiber makes it impossible, let it be 10 mm). Fix one by one both ends of a fiber using adhesives(5) in the manner that a fiber is loosely stretched across a section, and use this as a sample. Attach the sample to the grips of the tension tester for a single fiber and, after cutting the paper near an upper grip, carry out testing at either condition of those shown at Table 2.. Read the elongation as a looseness (mm) when loading with pre-tension and, the load gf{N} and elongation (mm) when the sample is broken due to increasing load. According to the following foumula, calculate a tensile strength (gf/D){N/tex} and elongation percentage (%). Carry out tests specified times(6), and express the average of them (to the second decimal place for the tensile strength, and to the first decimal place for the elongation percentage).

Tensile strength (gf/D) N/tex $= \frac{SD}{d}$

where SD: strength when broken (gf){N}

d: fineness of corrected weight of smaple (D) {tex}

Elongation percentage (%) $= \frac{E_2 - E_1}{L + E_1} \times 100$

where E_1: looseness (mm)

E_2: elongation (mm) when broken or elongation (mm) at maximum loading

L: distance between grips (mm)

Fig. 3

Unit: mm

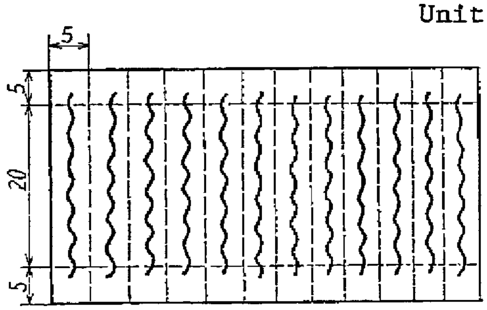


Table 2

Kind of tester	Distance of grips(7)	Tensile speed
Constant-rate tension type	20 mm	20 ± 1 mm/min.
constant-rate loading type	20 mm	The rate by which all capacity is loaded for one minute. Example: In the case of tester having capacity 50 gf {490 mN}, loading rate is 50 gf/min {490 mN/min}.
Constant-rate elongation type	20 mm	The rate at which 100 % or 50 % of grips distance is elongated per one minute.

Notes (5) Select suitable adhesive according to the kind of fiber, which shall give no damage to the fiber.

(6) In the case of rayon and cupro, carry out 50 tests times, and in the case of other than these, 30 times.

(7) When it is impossible to test by 20 mm because of the shortness of fiber, 10 mm is admissible.

Remarks 1. Carry out by using either one of abovementioned conditions tests, and append the kind, capacity, interval of grips, and rate of increasing tension of the tester used.

2. It is admissible to attach the sample to grips under the condition that pre-tension is loaded in the sample. In this case the looseness becomes 0 mm.

3. If necessary, carry out the test by controlling the time required until breaking to be 20 ± 3 s.

4. When the load at breaking is not the maximum load (or in other words, the load at breaking is smaller than the maximum), measure the maximum load and the elongation shown at that time.

5. If the fiber is broken at the spot being gripped, this test should be omitted.

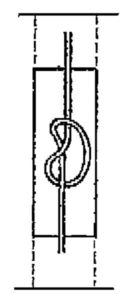
7.7.2 Test in Wet State Put the sample prepared in the way similar to 7.7.1 into another container and, after immersing it in water (20 ± 2°C) for two minutes to make it be sufficiently wet, measure and obtain tensile strength (gf) {N} and elongation percentage (%) in water(8) similarly to 7.7.1 (to the second decimal place).

Note (8) In the case of rayon, cupro, and acetate, immerse the sample again in water after loading pre-tension and reading the looseness on the sample taken out from water.

7.8 Knot Strength

7.8.1 Test at Standard State On glossy and smooth paper, fix one by one both ends of a fiber similarly to 7.7.1 in such a manner that the fiber is loosely stretched and a knot made previously is set at center of a section as shown in Fig. 4, and use this as a sample. Grip the sample under the same condition as 7.7.1 so as to position the knot at center of two grips, and measure the knot strength (gf) {N} at breaking, and express the average (to the second decimal place).

Fig. 4



Remark: If the fiber is broken at a place except knot when testing, this test shall be rescinded.

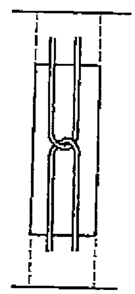
7.8.2 Test in Wet State Put the sample prepared similarly to 7.8.1 into another container, and after immersion in water (20 ± 2°C) for two minutes to dampen it sufficiently, measure and obtain the knot strength (gf) {N} in water similarly to 7.8.1 (to the second decimal place).

Remark: This is mainly applied to synthetic fiber.

7.9 Loop Strength

7.9.1 Test in Standard State On glossy and smooth paper, fix one by one both ends of a fiber similarly to 7.7.1 in such a manner as that the fiber is loosely stretched and the loop made previously by two fibers is set at center of section as shown in Fig. 5, and use this as the sample. Grip the sample under the same condition as 7.7.1 so as to position the knot at center of two grips, and measure and obtain the loop strength (gf){N} when broken, and express the average (to the second decimal place).

Fig. 5



Remark: If the fiber is cut at a place except loop when testing, this measure value shall be rescinded.

7.9.2 Test in Wet State Put the sample prepared similarly to 7.9.1 into another container, and after immersion in water (20 $\pm$ 2°C) for two minutes to make it sufficiently wet, measure and obtain the loop strength (gf){N} in water similarly to 7.9.1.(to the second decimal place).

Remark: This is mainly applied to synthetic fiber.

7.10 Modulus of Elongation Elasticity

Method A Fix the sample prepared similarly to 7.7.1([9]) to the grips of a constant-rate elongation type tension tester equipped with an automatic recording unit, and measure accurately the length shown at pre-tension. Next after letting the sample elongate by 3 %([10]) of the length owing to the pre-tension with the tensile rate 10 % and 50 % of grip interval per minute, immediately remove the load at the same rate. After standing for two minutes, again elongate the sample at the same rate until it attains a specified length. From the load-elongation curve (Fig. 6) recorded, measure a residual elongation, and calculate the modulus of elongation elasticity (%) according to the following formula. Carry out ten times the test, and express the average of them (by integer).

Modulus of elongation elasticity (%) $= \frac{l-l_1}{l} \times 100$

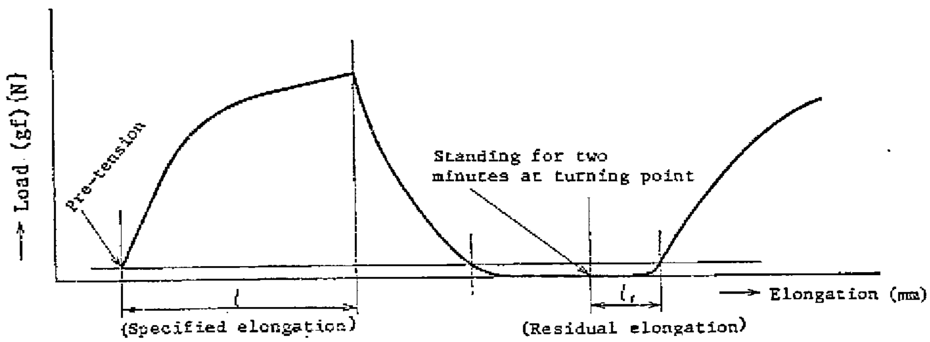
wwhere l : elongation when 3 % elongated (mm)

l_1 : residual elongation (mm)

Notes (9) If necessary, 50 mm of grip interval may be taken.

(10) Where required, 2 % or 5 % may be available instead of 3 % elongation.

Fig. 6



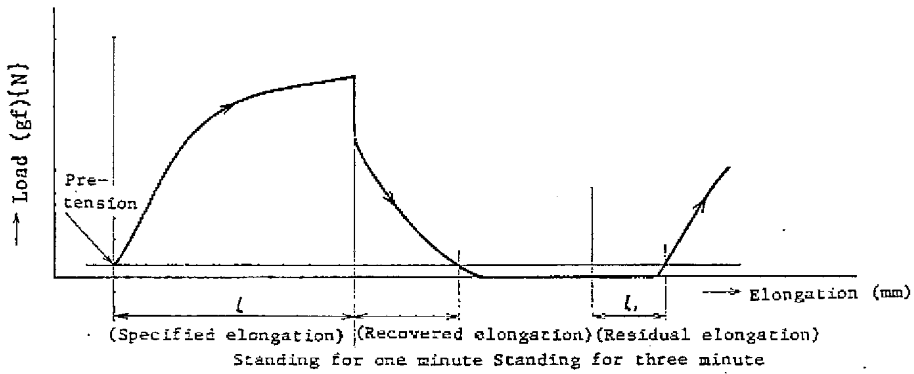
Method B Similarly to Method A, fix the sample to the grips and, after letting the sample elongate by 3 %(10) of the length due to the pre-tension with the tension rate 10 % or 50 % of the grip interval per minutes, allow it stand for one minute. Then, remove the load at the same rate and, after standing for three minutes, again elongate the sample at the same rate until it reaches specified length. From the load-elongation curve (Fig. 7) recorded, measure a residual elongation, and calculate the modulus of elongation elasticity (%) according to the following formula. Carry out five times the test, and express the average of them (by integer).

Modulus of elongation elasticity (%) $= \frac{l-l_1}{l} \times 100$

where l : elongation when 3 % elongated (mm)

l_1 : residual elongation (mm)

Fig. 7



Remarks 1. Append a note stating the kind of testing and testing condition (distance between grips, tensile rate, and rate of elongation).

2. It should be preferable that the load charged at the specified elongation becomes 50 % of the full scale of a recording paper at least.

3. The speed of a recording paper shall be set so that a specified elongation corresponds to the length of at least 5 cm on the recording paper.

4. Method A is applicable to all kind of fibers and Method B mainly to synthetic fibers.

7.11 Initial Tensile Resistivity After carrying out the test similarly to 7.7.1 on the sample, draw a load-elongation curve as shown in Fig. 8, and on the curve find the point A where the ratio of load change to elongation change is the maximum (the maximum point of tangent angle). Calculate the initial tensile resistivity (gf/D) {N/tex} according to the following formula. Carry out ten times the test, and express the average of them (by integer).

$$\text{Initial tensile resistivity (gf/D)\{N/tex\}} = \frac{P}{\frac{l'}{l} \times d}$$

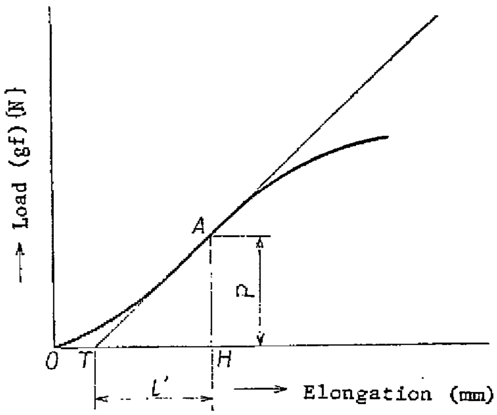
where P : load at A where tangent angle is the maximum (gf){N}

d : fineness based on corrected weight (D){tex}

l : length of sample tested (mm)

l' : length of TH (mm)
(H is the foot of a perpendicular and T is the intersecting point of a tangent and abscissa.)

Fig. 8



Remarks 1. In order to lessen the measuring error, the chart speed should preferably be controlled so that the tangent at A on the initial load and elongation curve may make about 45° angle to the axis of elongation.

2. The relation between the initial tensile resistivity and the apparent Young's modulus is as follows:

Apparent young's modulus (kgf/mm^2) {N/mm^2}
= 9 x ρ x inilital tensile resistivity (gf/D) {N/tex}
where ρ: density of fiber (g/cm^3)

3. Append a note stating the kind of a tester and the condition at tension test.

7.12 Crimp

7.12.1 Crimp Number On glossy and smooth paper on which sectioning lines have been drawn similarly to 7.7.1 (on condition that spacing distance is 25 mm, but in case it is impossible because of shortness of the fibers, it is taken as 20 mm), fix one by one the samples which are sampled from several parts where crimps are intact, preserved at both ends with adhesives(5) in the manner to let the fiber have 25 $\pm$ 5 % looseness to the strip in each of sections. Attach each of these fibers at the grips of a crimp tester, and, after cutting the strip, read the distance between grips (spacing distance) (mm) and count the number of crimps under loading pre-tension(11), and obtain the number of crimps per 25 mm. Carry out 20 times the test, and express the average of them (to the first decimal place).

Note (11) The pre-tension for the counting number of crimps should be 2 mgf{0.18 mN} per one denier {one tex} of marked denier {tex}. To vinylidene, however, 0.3 mgf {0.026 mN} per one denier {one tex} of marked denier tex shall be allotted.

Remarks 1. When reading the number of crimps, count the number of all peaks and bottoms, and divide it by 2.

2. When the distance of grips is made 20 mm, append a note stating this.

7.12.2 Crimp Percentage and Residual Crimp Percentage Measure the length when loading pre-tension(11) on the sample, and the length(12) when further loading(13) it. Then, after removing all loads and standing for two minutes, read the length when again loading pre-tension, and calculate the crimp percentage (%) and residual crimp percentage (%) according to the following formula. Carry out 20 times the test, and express the average of them (to the first decimal place).

$$\text{Crimp percentage (\%)} = \frac{b-a}{b} \times 100$$

$$\text{Residual crimp percentage (\%)} = \frac{b-c}{b} \times 100$$

where a : length when loading pre-tension (mm)

b : length when loading 50 mgf {4.41 mN } per 1 denier {1 tex }(mm)

c : after two-minutes standing, length when loading pre-tension (mm)

Notes (12) It should be 50 mgf {4.41 mN } per one denier {one tex } of marked denier tex . For vinylidene, however, it should be 25 mgf { 2.21 mN } per one denier {1 tex } of marked denier { tex }.

(13) Measure the length immediately after this in the case of rayon, cupro, and acetate, and 30 sec. later in the case of synthetic fibers.

Remark: When the loading is not suitable for the test, employ some or other suitable load, and append a note stating this.

7.12.3 Modulus Crimp Elasticity From the result in 7.12.2, calculate the modulus of crimp elasticity (%) according to the following formula (to the first decimal place).

$$\text{Modulus of crimp elasticity (\%)} = \frac{b-c}{b-a} \times 100$$

7.13 Coefficient of Friction After changing the sample into uniform sliver by opening sufficiently with a hand card, wind the fiber around the cylinder (8 mm in outside diameter) of a Roder-type friction coefficient tester to make the direction of fiber parallel to the axis of the cylinder. Next, pick up at random one fiber from the same sample and, after hanging the fiber, both ends of which are fitted with pre-tension(14), at the center of a cylindrical sliver, connect one end of the fiber to the hook of a tortion balance.

For measuring static friction coefficient (μs), stop the cylindrical sliver and obtain the load when the balance between both sides is lost, by means of a tortion balance. For dynamic friction coefficient (μd), revolve the cylindrical sliver at circumferential speed of 90 cm/min and, after obtaining the load when both sides regain blancing by means of a tortion balance, calculate the friction coefficient according to the following formula:

Carry out the 30 times in total, that is, three cylindrical slivers are tested with respectively ten test fibers, and express by the average of them (to the third decimal place).

Coefficient of friction (μs or μd) $= 0.733 \log \frac{W}{W-m}$

where, W: load at both ends of fiber (mgf) {N}

m: reading of tortion balance (mgf) {N}

Note (14) When the loading is not suitable for the test, use some or other suitable load, and append a note stating this.

Remark: When applying different testing conditions, append a note.

7.14 Specific Gravity and Density

7.14.1 Specific Gravity (Sink-and-Float Method) Place about 0.1 g of the sample in a beaker under the condition of being free from any tension, and treat it with about 20 ml of de-oiling liquid(15) for about one hour. After de-oiling, air-dry the sample and, after cutting it into 0.5 mm to 1.0 mm by using a razor edge or the like, dry in a vacuum desiccator(16) to an absolute dry condition. Put about 8 ml of prepared specific gravity measuring liquid(17) in a precipitation tube, and place a little of the sample. After stoppering, disperse the sample sufficiently and, after removing bubbles by means of a centrifugal separator, immerse it in a thermostatic bath of 20.0 ± 0.1°C temperature, which is let to stand for about 30 minutes. Observing carefully the sink-and-float condition of the sample, control the specific gravity of the liquid to get an equilibrium state of the sample in the liquid in a precipitation tube by means of adding suitable light liquid or heavy liquid into the specific gravity measuring liquid. After adjusting, let the liquid stand further 30 minutes in the thermostatic bath, and again confirm the equilibrium condition of the sample. After confirming, measure the specific gravity (to the third decimal place) of the liquid according to 3.2.2 Second Method in JIS K 0061, and take it as the specific gravity of the sample (d_4^{20}).

Notes (15) As suitable solvents for de-oiling the adhering substances of the sample, the following de-oiling liquids or the equivalent solvents shall be used.

Table 3

Target fibers	De-oiling liquid
Polyester	Methanol
Acetate	Diethyl either
Polypropyrene	Ethanol
Others	Mixture of ethanol and benzene (1:2 by volume ratio)

Notes (16) Make the vacuum degree 4 mm Hg or lower.

(17) After mixing suitably light liquid and heavy liquid which correspond to measuring fibers, the specific gravity of the mixture should be controlled so as to fall in the range of $\pm$ 0.02 from the specific gravity of target fiber by using a Baume's hydrometer.

Table 4

Target fibers	Light liquid	Heavy liquid
Polypropylene	Water	Ethanol
Polyester	Carbon tetrachloride	*n*-heptane
Others	Carbon tetrachloride	Toluene

Remark: The tools used for the tests shall be in accordance with JIS K 0050.

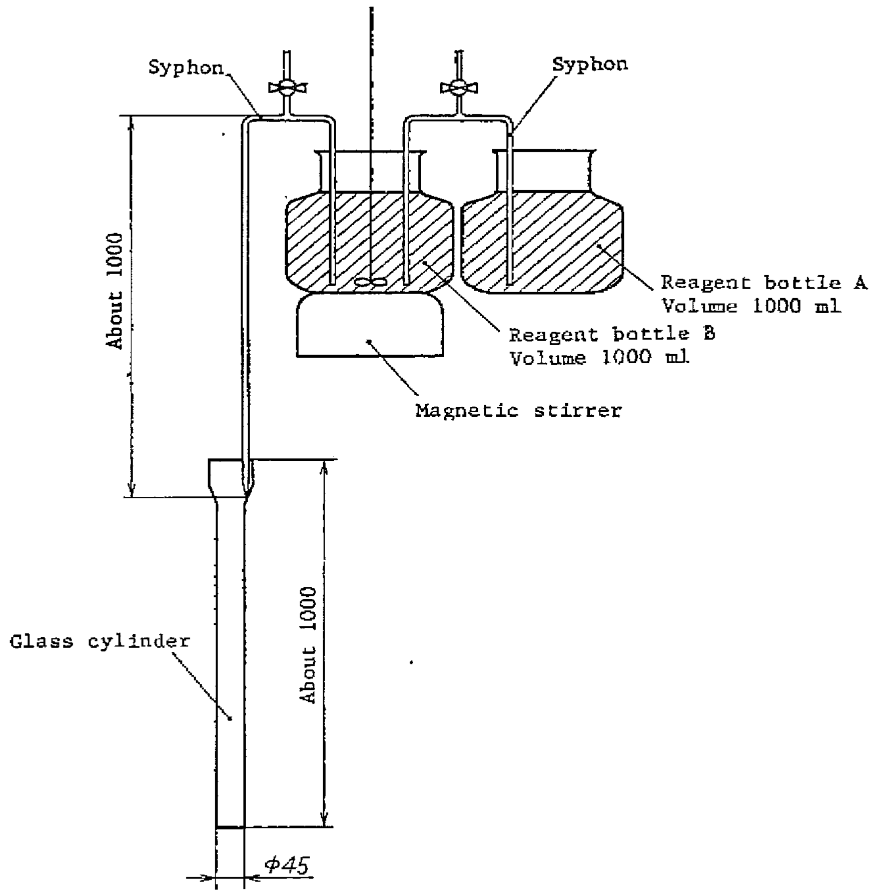
Reagents shall be the guaranteed ones specified in the following JISs: JIS K 8891, JIS K 8102, JIS K 8103, JIS K 8858, JIS K 8459, JIS K 8680.

7.14.2 <u>Density (Density Gradient Tube Method)</u> By using the apparatus shown in Fig. 9, after placing low density liquid(18) prepared previsouly in a reagent bottle A and high density liquid(18) in a reagent bottle B, connect two bottles that are leveled with a syphon. While stirring the liquid in B with a magnetic stirrer, let the liquid in B flow in a glass cylinder by letting it run on a wall at a rate of 10 ml/min or less. As the liquid level in the bottle is lowered, the liquid in the bottle A flows into the bottle B continuously. The liquid subsequently flows into the glass cylinder, so that the liquid in the cylinder shows continuous density gradient. Immerse quietly the glass cylinder filled with liquid into a thermostatic bath. Then, put a standard float(19), after being wetted with liquid in the bottle A (low density liquid), in the glass cylinder

and, keep this cylinder, referred to as a density gradient tube, in the thermostatic bath of 20 $\pm$ 0.1°C. After 24 hours, plot a correction curve([20]) between the density of the standard float in the tube and the readings of the density gradient tube. After the sample of about 0.1 g is deoiled similarly to 7.14.1, tie the sample that has been air-dried into a ring of 3 mm in diameter. Degasify the sample ([21]) in 5 to 6 ml of low density liquid, and by using twezers transfer it quietly into the density gradient tube. After the sample has stood at an equilibrium position in the liquid, read the depth shown by the sample to the nearest 1 mm by the scale on the density gradient tube. Comparing the value read on the scale with the correction curve, obtain the density (g/cm^3). Carry out two times the test, and express the average of them (to the third decimal place).

Fig. 9. Device of Density Gradient Tube

Unit: mm



Notes ([18]) High density liquid and low density liquid shall be prepared by mixing the light liquid and heavy liquid corresponding respectively to measuring fibers with the mixing ratio calculated according to the following formula. Prepare the tube so as to make the density discrepancy between the uppermost layer and the lowermost layer (whose density is the same as that of heavy liquid) 0.5 to 0.8 g/cm^3, and adjust the density of central layer liquid to correspond to that of target fiber.

Table 5

Target fibers	Light liquid	Heavy liquid
Polypropylene	Water	Ethanol
Polyester	Carbon tetrachloride	n-heptane or ligroin
Others	Carbon tetrachloride	n-heptane or toluene

Preparation of High Density Liquid

Quantity of heavy liquid used (ml): $\frac{\rho_1 - d_2}{d_1 - d_2} \times V_0$

Quantity of light liquid used (ml): V_0 − (Quantity of heavy liquid used)

Preparation of Low Density Liquid

Quantity of heavy liquid used (ml): $\frac{\rho_2 - d_2}{d_1 - d_2} \times V_0$

Quantity of light liquid used (ml): V_0 − (Quantity of heavy liquid used)

where ρ_1 : specific gravity of high density liquid

ρ_2 : specific gravity of low density liquid

d_1 : specific gravity of heavy liquid

d_2 : specific gravity of light liquid

V_0 : volume of high density liquid or low density liquid (ml) prepared, which is calculated according to the following formula:

$$V_0 = \frac{\text{Liquid volume in density gradient tube (ml)}}{2} \times \frac{\rho_1 - \rho_2}{\rho_1 - \rho_2'}$$

(ρ_2' is the specific gravity of the uppermost layer in the tube.)

([19]) This is a hollow glass sphere 3 to 5 mm in diameter, and one or more should be used per 0.01 g/cm^3 of density difference.

Notes (20) When the correction curve shows a zigzag shape or serious bow shape, repeat the operation from the beginning.

(21) Degasify either by placing the sample under reduced pressure at 5 mm Hg for 5 minutes as it is kept in low density liquid, or subjecting the sample to a centrifugal separator of 2000 to 3000 rpm for 2 to 3 minutes.

Remark: The tools used for the tests shall be in accordance with JIS K 0050.
Reagents shall be the guaranteed reagent specified in the following JIS:

JIS K 8102, JIS K 8459, JIS K 8937, JIS K 8680.

7.15 Shrinkage Percentage

(1) Shrinkage percentage by Hot Water On the glossy and smooth paper on which sectioning lines have been drawn similarly to 7.7.1 (on condition that spacing distance is 25 mm, but in case shortness of the fibers makes it impossible, it 20 mm, but in and in the case of length enough to be tested by 50 mm, it shall be 50 mm). Fix one by one the fibers at both ends with adhesives(22) in the manner to keep the fiber stretched loosely in each of sections to form the sample. Attach the sample to a suitable suspending tester with the grip distance of 25 mm (when making the spacing distance 20 mm or 50 mm, the grip distance should be respectively 20 mm or 50 mm) and, after cutting the paper, read the distance (mm) between two grips under loading pre-tension. Wrap the sample unfixed from the tester in a suitable cloth(23) and, after immersing in hot water of suitable temperature(24) for 30 minutes, draw out the smaple. After taking away softly the water with a blotting paper or cloth and after air-drying, attach again the sample to the tester, and read the distance (spacing distance of the sample) (mm) between grips when loading it with pre-tension. Calculate the shrinkage by hot water (%) according to the following formula. Carry out 30 times the test, and express the average of them (to the first decimal place).

Shrinkage percentage by hot water (%) $= \frac{L-L'}{L} \times 100$

where L : distance between grips when loading pre-tension before treatment (mm)

L' : distance between grips when loading pre-tension after treatment (mm)

Notes (22) Use the adhesives that do not corrode the fiber and are endurable to hot water.

(23) Nylon taffeta is applicable.

(24) Append a note stating the temperature applied.

(2) Shrinkage Percentage at Hot Dry Prepare the sample similarly to the case of (1) shrinkage percentage by hot water, and read the distance (mm) between grips when loading pre-tension. Suspend the sample after unfixing from the tester in a dryer at suitable temperature(24), and take out after standing for 30 minutes. After cooling to room temperature, load again with pre-tension, and read the distance between grips (spacing distance of the sample) (mm). Calculate the shrinkage percentage at hot dry (%) according to the following formula. Carry out 30 times the test, and express the average of them (to the first decimal place).

$$\text{Shrinkage percentage at hot dry (\%)} = \frac{L-L'}{L}\times 100$$

where L: distance between grips when loading pre-tension before treatment(mm)

L': distance between grips when loading pre-tension after treatment (mm)

Remark: Append a note stating the kind of testing method and conditions (distance between grips and thermal treatment temperature).

7.16 Melting Point and Thermal Shrinkage Temperature

7.16.1 Melting Point

Method A Use a microscope with a stage equipped with a polarizing device and heating apparatus, and make its visual field dark black by intersecting rectangularly a polarizer and an analyzer. After putting the sample on a slide glass, set at 45° the angle between the axis of the sample and both the vibration idrections of light of the polarizer and analyzer, and now, the crystalline part of the sample is glittering and other parts become black. Heat the stage at a rate of 1°C rising per minute from about 10°C lower than the melting point. Because the glittering disappears due to the melting of crystal line part, take this point as melting point. Carry out three times the test and express the average of them (by integer).

Method B After putting the sample in a capillary, heat the sample in a heating apparatus at a rate of 1°C rising per minute from 10°C lower than the melting point, and read the temperature when the sample melts. Carry out 3 times the test and record the average of them (by integer).

Remarks 1. Correct the thermometer on a heating apparatus.

2. Carry out the measurement of melting point mainly for nylon, polyester, and polypropylene, and append a note stating the measuring method.

7.16.2 Thermal Shrinkage Temperature Suspend the sample applied specified load(25) in a measuring tube for the melting point (Fig. 10) or in a thermostatic dryer(26), and heat gradually the sample at a rate of 1°C rising per minute from about 10°C lower than the melting point.

Set the length of the smaple at 2 to 10 cm, and read the temperature when the sample gets a specified shrinkage or the maximum shrinkage(27). Carry out 3 times the test, and express the average of them (by integer).

Notes (25) Specified loads mean the following numerical values:

Vinylon .... 2 mgf{0.18 mN}per 1 D{1 tex} of nominal denier {tex}

Acrylic, modacrylic, polychlal ... 5 mgf {0.44 mN}per 1 D {1 tex}of nominal denier{tex}

Polyvinyl chloride ..... 10 mgf{0.88 mN} per 1 D {1 tex} of nominal denier tex

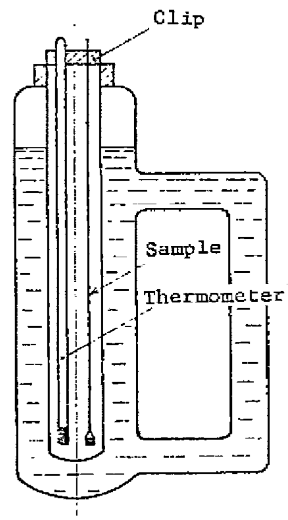
Vinylidene .... 1 mgf{0.088 mN} per 1 D {1 tex} of nominal denier {tex}

(26) This shall be capable of being observed through visually and equipped with a voltage regulator.

(27) Measure the temperature when 10 % shrinkage is observed in cases of vinylon, polyvinyl chloride, and polychlal, and when the shrinkage is the maximum in cases of acrylic, modacrylic, vinylidene; and when measuring at other temperatures than the above, append a note stating it.

Remark: Carry out the test of thermal shringkage temperature mainly for vinylon, polyvinyl chloride, acrylic, modacrylic, vinylidene, and polychlal, and append a note stating the testing condition.

Fig. 10



7. 17 Whiteness After changing the sample into a uniform sliver by combing sufficiently with a hand card, pack the sample in a case at random without arranging fiber direction as far as possible, so as to pack 0.15 x ρ g [ρ means the density of fiber (g/cm³)] per 1 ml of the case. Cover closely the front of the sample and standard white surface([28]) with a flat, uniform, and transparent glass plate about 1 mm in thickness. Measure by using either one of the following methods, and append a note stating the method applied. Carry out the test two times in the case of Method A and Method D, and 5 times in the case of method B and Method C, and express the average of them (with two significant figures).

Note ([28]) Standard white surface shall be in accordance with 4.3.4 in JIS Z 8722.

Method A (Judd's Method) By means of the spectrophotometer equipped with the reflecting light measuring device by an integrating sphere, measure the characteristic curve on spectro-reflectivity to the standard white board with the range of visible rays (400 to 700 nm).

Calculate three stimulus values X, Y, and Z from the characteristic curve on spectro-reflectivity and, after obtaining x and y([29]) respectively as chromaticity coordinates, calculate a white index (W) according to the following formula. Represent the whiteness by using this W.

$$W=1-[\{30(\alpha^2+\beta^2)^{\frac{1}{2}}\}^2+\{(1.00-Y)/2\}^2]^{\frac{1}{2}}$$

where $\alpha : \dfrac{2.4266x-1.3631y-0.3214}{1.0000x+2.2633y+1.1054}$

$\beta : \dfrac{0.5710x+1.2447y-0.5708}{1.0000x+2.2633y+1.1054}$

Note ([29]) The calculation of X, Y, Z, x, and y shall be in accordance with JIS Z 8701.

Method B (Two-Wavelength Method) By using a trichromatic colorimeter, calculate the whiteness according to the following formula from the data of B(30) (reflectivity on blue) and G(30) (reflectivity on green).

$$\text{whiteness} = 4B - 3G$$

Note (30) Combination of the light source of a trichromatic colorimeter, a filter, and a light receiver should preferably satisfy Luther conditon.

Remark: Instead of B and G, the reflectivities at wavelength 450 nm and 550 nm measured by a spectrophotometer may be applied.

Method C (Hunter Method) By using a hunter color difference meter, measure L, a, and b, and calculate the whiteness according to the following formula:

$$\text{Whiteness} = 100 - [(100-L)^2 + a^2 + b^2]^{\frac{1}{2}}$$

Remark: L, a, and b may be calcualted from the tristimulus values X, Y, and Z by using the following formula:

$$L = 100Y^{\frac{1}{2}}$$

$$a = 175(1.02X - Y)/Y^{\frac{1}{2}}$$

$$b = 70(Y - 0.8472)/Y^{\frac{1}{2}}$$

Method D (Easy Method - Definite Wavelength Method) By means of a spectrophotometer equipped with a reflecting light measuring device by an integrating sphere, or a photoelectric photometer, measure the reflectivity R (%) to a standard white board on the wavelength of 480 nm(31), and represent the whiteness by using this R.

Note (31) In the case of a photoelectric photometer, the filter of 480 nm in main wavelength and 30 nm in width of wavelength shall be used.

Remark: Preparation of Standard White Board Burning gently purified magnesium, let white fume deposit uniformly on a flat white board to get the thickness of 1 mm or more.

7.18 Glossiness After changing 0.3 g of the sample into a uniform sliver by combing sufficiently with a hand card, on a black velvet board of 45 mm^2, arrange the whole sample by applying pressure in paralle so as to make its surface uniform. Next, putting the sample board on a rotating stage of a Pulfrich photometer and pressing the board by a shutting frame, attach it so as to let the direction of the fibers face to a light source when the directing angle of the stage (δ) is zero, and on the other side attach a standard white board. Using a L_2 filter (central wavelength of a filter is 540 to 550 nm), calculate the glossiness (η) by using the following formula from the data of the luminance H_0 in cases where rotating angle δ is zero and the luminance H_1 in cases where rotating angle is 22.5°. Carry out the test twice, and express the average of them (to the second decimal place).

Glossiness $(\eta)=\frac{H_1}{H_0}K\delta$

where H_0 : luminance of sample at standard position $(\delta=0°)$

H_1 : luminance of sample at rotated position $(\delta=22.5°)$

$K\delta$: correction factor of glossiness on standard white board

Remarks 1. $K\delta$ (22.5°) should be 1.037.

2. Preparation of Standard White Board Burning gently purified magnesium, let white fume deposit uniformly on a flat white board to get the thickness of 1 mm or more.

3. A tester having performance equivalent to a Pulfrich photometer may be used.

7.19 Abnormal Fibers Out of 500 g of sample, select the abnormal fibers caused by such as gum knot, insufficient drawing, and others, and thus determine the content percentage by the number of mg per 100 g sample (to the first decimal place).

Remark: The criteria of selecting speed per 100 g of the sample shall be 30 minutes for layon and acetate, and 15 minutes for synthetic fibers.

7.20 Ash Content Weigh accurately 5 to 10 g of the sample whose water content has been known into a crucible and, after burning gradually with care lest the sample should scatter, heat to incinerate it for two hours at about 850°C. Weigh its mass after cooling in a desiccator. Then, ignite again for 30 minutes and weigh it. Repeat this operation until the discrepancy of two weights becomes 0.5 mg or less, and calculate the ash content (%) according to the following formula. Carry out two times the test, and express the average of them (to the second decimal place).

Ash content (%) $= \frac{W'}{W} \times 100$

where W: absolute dry mass of sample (g)

W': mass of residue after ignition (g)

7.21 Titanium Oxide Weigh accurately about 5 g of the sample whose water content has been known, and heat to incinerate in an electric furnace avoiding ignition. Transfer the ash into a 200 ml beaker with a little of water. After heating the beaker to evaporate the water, add 15 ml of concentrated sulfuric acid (sp. gr. 1.84)([32]) and 10 g of ammonium sulfate([33]) and, after covering with a watch glass, heat on a sand bath at first gradually and finally strongly to get a transparent liquid. After cooling, add water up to about 100 ml lest the liquid temperature should rise to higher than 50°C, and transfer into a 1 l measuring flask to dilute with water up to the marked line. From the solution, pipet Aml ([34]) aliquot into a 50 ml measuring flask, and after coloring by adding 5 ml of 3 % hydrogen peroxide water ([35]) and 10 ml of 2 N sulfuric acid, dilute with water up to the marked line. Measure the absorbance of this liquid, which is kept in a cell, by using a photoelectric colorimeter at wavelength of 420 nm, and obtain the concentration of titanium oxide (g/50 ml) by using a working curve prepared previously. Calculate the percentage of titanium oxide according to the following formula. Carry out twice this test, and express the average of them (to the second decimal place).

Titanium oxide (%) $= \frac{B \times 1000}{C \times A} \times 100$

where A : volume of aliquot from diluted solution (ml)

B : concentration of titanium oxide (g/50 ml)

C : absolute dry mass of sample (g)

Notes ([32]) Use guaranteed reagent specified in JIS K 8951.

([33]) Use guaranteed reagent specified in JIS K 8960.

([34]) The volume of aliquot shall be enough to make the absorbance of colored solution 0.3 to 0.5 according to the content of titanium oxide and the thickness of a cell.

([35]) Use guaranteed reagent specified in JIS K 8230.

Remarks 1. Preparation of Working Curve To prepare titanium oxide standard solution, weigh accurately about 0.5 g of titanium oxide under absolute dry condition whose purity has been known, and treat it similarly to the case of the sample by adding 50 ml of concentrated sulfuric acid (sp.gr. 1.84) and about 35 g of ammonium sulfate. Transfer into a 1 l measuring flask, and dilute it with water up to the marked line.

Pipet four or more aliquots, which are different volumes from each other, from the titanium oxide standard solution [volume satisfying the condition shown in Note ([34])] into 50 ml flasks, and after treating them similarly to the sample, find the absorbances to prepare a working curve.

Check periodically the working curve.

In case of alternation of reagents or a color comparing apparatus, renew the working curve.

2. The appliances used in the tests shall be in accordance with JIS K 0050.

7.22 Solvent Extractable Matter Carry out test by either one of the following methods, and append a note slating the method adopted.

(1) Washing Loss Weigh accurately about 5 g of the sample whose water content has been known into an Elrenmeyer flask together with about 0.5 % nonionic surface active agent of 100 times quantity as much as the sample, and treat it for 30 minutes while stirring at 40 ± 2°C. Filtrate it through a funnel and, after washing the sample sufficiently with warm water and after drying, weigh the mass dried absolutely. Calculate the washing loss (%) according to the following formula. Carry out twice the test, and express the average of them (to the second decimal place).

$$\text{Washing loss (\%)} = \frac{W-W'}{W} \times 100$$

where W : mass of sample (g)

W' : absolute dry mass of sample washed (g)

Remark: This is applicable mainly to synthetic fibers.

(2) Extract by Alcohol and Benzene Weigh accurately 5 g of the sample whose water content has been known into loosely a Soxhlet extractor([36]) with no filter paper thimble, and pour 100 to 150 ml of the mixture of alcohol and benzene([37]) (Volumetric ratio is 1:2) into an attached flask. Heat it on a water bath for 3 hours keeping the state of gently boiling([38]) the extracting mixture, and then transfer back the mixture gathered in the sample container into the flask. After concentrating the solution in the flask to 5 ml or less (if necessary, filtrate through a sintered glass filter of 1G1 or 3G1), transfer it into a weighing bottle whose constant weight at 105 ± 2°C has been obtained previously. Wash the extracting flask with hot alcohol and benzene mixture, and pour washings (in the case of using a sintered glass filter, after filtrating the washings through one of the above-mentioned sintered glass filter) into the weighing bottle. Then, after evaporating the solvent on a water bath, let it stand alone for 1.5 hours in a thermostatic dryer at 105 ± 2°C, and weigh the mass after cooling in a desiccator. The content of extract shall be expressed by the percentage of the extract produced by the mixture to the absolute dry mass of sample. Carry out twice the test, and express the average of them (to the second decimal place).

Notes ([36]) The Soxhlet extractor specified in JIS R 3503 shall be used.

([37]) Use the guaranteed grade reagents specified in JIS K 8102 and JIS K 8858. When using mixture solution alcohol and benzene that has been recovered, use it after making the water content 1.7 ± 0.5 %.

([38]) Heat the extractor to such an extent as the solvent refluxes through a syphon tube once per 10 minutes.

Remark: This is applied mainly to rayon, cupro, and synthetic fibers.

(3) Extract by Diethyl Ether Weigh accurately 5 g of the sample whose water content has been known, transfer it into a Soxhlet extractor(36) with no filter paper thimble, and put 100 to 150 ml of diethyl ether(39) into an attached flask. Heat it on a water bath for 1.5 hours keeping the state of gently boiling(38) the extracting solution, and then transfer back the extracting solvent gathered in the sample container into the flask. After concentrating the solution in the flask to 10 to 15 ml (if necessary, filtrate through a sintered glass filter of 1G1 or 3G1), transfer it into a weighing bottle whose constant weight at 105 ± 2°C has been obtained previously. Wash the extracting flask with diethyl ether, and pour washings (in the case of using sintered glass filter, after filtrating the washings through the above-mentioned sintered glass filter) into the weighing bottle. Then, after evaporating the solvent on a water bath, let it stand alone for 1.5 hours in a thermostatic dryer at 105 ± 2°C, and weigh its mass after cooling in a desiccator. The content of extract shall be expressed by the percentage of the extract by diethyl ether to the absolute dry mass. Carry out twice the test, and express the average of them (to the second decimal place).

Note (39) Use the guaranteed grade reagent specified in JIS K 8103.

Remark: This is applied mainly to acetate and synthetic fibers.

(4) Extract by Carbon Tetrachloride Weigh accurately about 5 g of the sample whose water content has been known into a suitable vessel and after adding 150 ml of carbon tetrachloride(40) that is dehydrated and cooled, dip it for 5 minutes at 20 ± 2°C by constantly stirring. Flow out the extracting solution into a 500 ml Elrenmeyer flask and, after washing twice the vessel with 150 ml of carbon tetrachloride, concentrate the extracting solution and the washings to make 5 ml or less (if necessary, filtrate through a sintered glass filter of 1G1 or 3G1). Transfer it into a weighing bottle whose constant weight has been obtained at 105 ± 2°C. Wash the extracting flask with carbon tetrachloride, and pour the washings into the weighing bottle (in the case of using a glass filter, after filtrating the washings through the same glass filter). After evaporating the solvent, let it stand for one hour in a thermostatic dryer at 105 ± 2°C, and weigh its mass after cooling in a desiccator. The content of extract shall be expressed by the percentage of the extract by carbon tetrachloride to the absolute dry mass of sample. Carry out twice the test, and express the average of them (to the second decimal place).

Note (40) Use the guaranteed grade reagent specified in JIS K 8459.

Remark: This is applied mainly to synthetic fibers.

(5) Extract by Methanol Weigh accurately about 5 g of the sample whose water content has been known, transfer it into a Soxhlet extractor([36]) with no extraction thimble, and pour 100 to 150 ml of methanol([41]) into an attached flask. Heat it on a water bath for 3 hours keeping the state([38]) of gently boiling the extracting solution, and then transfer back the extracting solvent gathered in the sample container into the flask. After concentrating the solution in the flask to 5 ml or less (if necessary, filtrate through a sintered glass filter of 1G1 or 3G1), transfer it into a weighing bottle whose constant weight at 105 ± 2°C has been obtained previously. Wash the extracting flask with methanol, and pour washings (in the case of using a sintered glass filter, after filtrating the washings through the same glass filter) into the weighing bottle. Then, after evaporating the solvent on a water bath, let it stand alone for 1.5 hour in a thermostatic dryer at 105 ± 2°C, and weigh its mass after cooling in a desiccator. The content of extract shall be expressed by the percentage of the extract by methanol to the absolute dry mass of example. Carry out twice the test, and express the average of them (to the second decimal place).

Note ([41]) Use the guaranteed grade reagent specified in JIS K 8891.

Remark: This is applied mainly to synthetic fibers.

Remark: The appliances used in the tests shall be in accordance with JIS K 0050.

7.23 Tensile Strength at Wet Condition after 5 % Alkali Treatment

Weigh out about 3 g of the sample, and dip it in a 1 *l* beaker in which 5.0 ± 0.1 % sodium hydroxide solution([42]) of 100 times quantity as much as the sample has been placed. After dipping for 5 minutes at 20 ± 1°C without any tension, immediately add the same quantity of water at same temperature by shaking gently. By means of either a decantation method or a Buchner funnel([43]), wash it with water at 30 to 40°C until it becomes almost neutral([44]), which is tested with phenolphthalein solution([45]) as indicator. Then dip the sample in 0.5 % acetic acid solution([46]) of 100 times quantity as much as the sample for about 5 minutes, and wash it similarly with water until showing no red color([47]) by the indicator of Methyl Orange solution([48]).

Separately treat similarly about 3 g of the sample, and render these two samples into water content equilibrium. Take 50 fibers in total, that is, 25 fibers from each of two samples, and calculate the tensile strength similarly to the description in 7.7.1.

When calculating, use the fineness based on corrected weight obtained in 7.5.1.

Notes ([42]) Use the guaranteed grade reagent specified in JIS K 8576.

([43]) Use a Buchner funnel measuring 70 mm in mouth diameter and 40 mm in depth. When washing by using the Buchner funnel, suck gently without using shirting.

([44]) Use the guaranteed grade reagent specified in JIS K 8799.

([45]) This means a state in which no coloring is seen any longer after the direct pouring of indicator into the solution.

([46]) Use the guaranteed grade reagent specified in JIS K 8355.

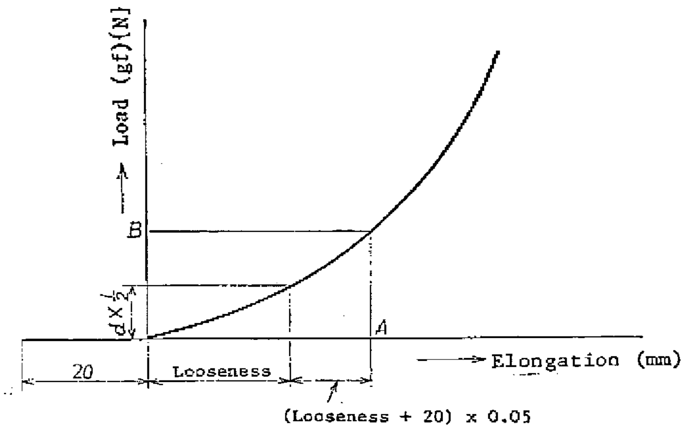
([47]) Use the guaranteed grade reagent specified in JIS K 8893.

([48]) This means a state in which coloring of yellow is shown by direct pouring of indicator on sample dried after treating.

Remark: this is applied to rayon and cupro.

7.24 5 % Elongation Stress at Wet Condition Apply a constant-rate elongation type tension tester equipped with an automatic recording unit similarly to 7.7.2 to the sample, and draw the curve of the load at wet condition and elongation under the tension rate of 10 mm per minute (Fig. 11). From this curve, obtain the elongation corresponding to 1/10 gf {8.84 mN} per 1 D {1 tex} of nominal denier nominal{tex}, and make this value as a looseness. Add this looseness to 5 % of the total length of this looseness and 20 mm, and then obtain on the curve the load (B) corresponding to the elongation (A) that is equal to the added length. Take the load (B) as stress, and express the stress by the number of grams gf/D {N/tex} per 1 D {1 tex} of the fineness of corrected weight. Carry out 20 times this test, and express the average of them (to the second decimal place).

Fig. 11



Remark: This is applied to rayon and cupro.

7.25 <u>Degree of Water Swelling</u> After combing sufficiently with a hand card, weigh about 2 g of the sample, and dip in 200 ml of water at 20 ± 2°C. After standing for 15 minutes in a thermostatic bath at 20 ± 2°C, squeeze softly and dehydrate it by using a centrifugal separator(49). In this case, put the sample in a separating tube in flatly spreaded condition. Apply 1000 to 1050 G of centrifugal force for dehydration and, after reaching specified revolution, switch off 10 minutes ± 10 seconds later then, let it tand to stop. After stopping, weigh the mass of the sample dehydrated.

Next, obtain the absolute dry mass of this sample, and calculate the degree of water swelling (%) according to the following formula. Carry out the test four times, and express the average of them (by integer).

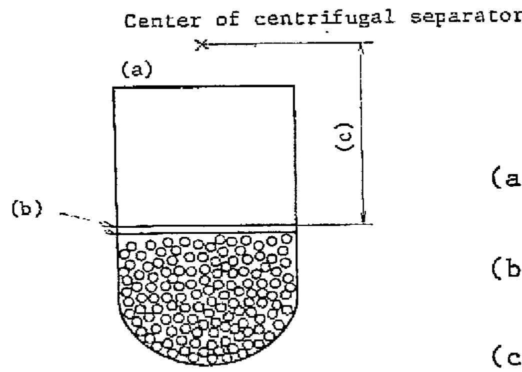
$$\text{Degree of water swelling (\%)} = \frac{W - W'}{W'} \times 100$$

where W : mass of sample dehydrated (g)

W' : absolute dry mass of sample (g)

Note (49) The centrifugal separator shown in Fig. 12 should preferably used.

Fig. 12



(a) Covering by polyethylene sheet

(b) Stainless steel net 2380 μm, 2 sheets

(c) When placing 105 mm apart, the revolution of the separator is 3000 rpm.

Remark: This is applied to rayon and cupro.

7.26 <u>Degree of Alkali Swelling</u> After combing sufficiently with a hand card, weigh out about 2 g of the sample, and dip in 200 ml of 25 $\pm$ 0.2 % sodium hydroxide solution ([42]) at 20 $\pm$ 2°C. After standing for 5 minutes in a thermostatic bath at 20 $\pm$ 2°C, squeeze gently with G2 filter and, after removing the liquid components similarly to 7.25 by using a centrifugal separator, weigh the mass of the sample from which the alkali liquid had been removed.

Next, wash this sample with 0.5 N acetic acid solution ([46]) until showing no alkaline property and, after washing the sample transferred on a glass filter with about 1 l of water and drying, obtain the absolute dry mass. Carry out four times the test, and express the average of them (by integer).

$$\text{Degree of alkali swelling (\%)} = \frac{W - W'}{W'} \times 100$$

where W: mass of sample freed from alkaline liquid (g)

W': absolute dry mass of sample (g)

Remark: This is applied to rayon and cupro.

7.27 <u>Skin Ratio</u> After combing and arranging sufficiently with a hand card, make them a bundle composed of about 50 fibers. After burying it in the mixture of ethyl cellulose, paraffin, and stearic acid([50])([51]), cut it rectangularly to the fiber axis by using a microtome by about 5 μm thickness in the manner to leave a part of outside layer of the burying material, so that several cuttings make a continuous stringlike slice. Put the continuous stringlike slice on the slide glass spread with mixture of the white of an egg and glycerin([52]), and heat gently until the slice sufficiently elongates and then sticks on it. Next, immerse this slide glass in the equi-volume mixture of xylene([53]) and ethanol([37]), and then, transfer

it from highly concentrated ethanol to lower ethanol([54]). Next, drop on the slices the dye liquor of azinebrilliant 5 blue Rconc([55]), and heat gently for 5 minutes on a hot plate of 100°C([56]). After removing the superfluous dye liquor from the slice with water rinsing, transfer the slice from low concentrated ethanol to higher concentrated ethanol in the reverse direction to the above-mentioned. After immersing the equi-volume mixture of xylene and ethanol, seal the slice with Canada balsam. Observe the sliced sample with a microscope and, after, if necessary, photographing or scaling up by drawing on a graph paper, obtain the area of both the section of fibers and skin layer. Calculate the skin ratio according to the following formula, and express the average of 20 fibers (by integer).

$$\text{Skin ratio (\%)} = \frac{H}{F} \times 100$$

where H: area of skin layer (mm^2)

F: sectional area (mm^2)

Notes ([50]) For the mixture, the mixing ratio of 0.5: 7: 3 is suitable.

([51]) Paraffin having melting point of 60 to 70°C, and stearic acid of 68°C or more are used.

([52]) The mixture of the white of an egg and glycerin is prepared as follows; mix the white of egg and glycerin with the ratio of 1: 1, and then add 1 % of sodium salicylic acid solution.

([53]) Use the guaranteed grade reagent specified in JIS K 8271.

([54]) The concentration of ethanol and dipping time shall be as follows:

(a)	Ethanol:	100	30 minutes
(b)	Ethanol:	water 90:10	30 minutes
(c)	Ethanol:	water 75:25	3 minutes
(d)	Ethanol:	water 50:50	3 minutes

([55]) Dissolve 3 g of dyestuff azinebrilliant blue 5 Rconc in 90 g of water by heating. After standing this solution for one day and night, filtrate through a sintered glass filter G4, and add 1 g of sodium sulfate and water up to 100 g.

([56]) Be careful lest the dye liquor should evaporate to dryness.

Remarks 1. The appliances used for testing shall be in accordance with JIS K 0050.

2. This is applied to rayon.

7.28 Sulfur Content Weigh out accurately about 5 g of the sample whose water content has been known, and after dipping it in a warm water at 70°C for about 5 minutes, wash it by sometimes stirring to mix. Repeat twice this operation and dehydrate. Transfer it into a 200 ml beaker. Add 2 ml of magnesium nitrate solution(57), 50 ml of nitroc acid(58) (60 %), and 5 ml of perchloric acid(59) (60 to 70 %), and covering with a watch glass, dissolve the sample by gently heating on a sand bath. When white fume of perchloric acid begins to rise after nitrogen dioxide gas finishes generating, wash the the adherings on the watch glass, and heat carefully and continuously to evaporate to dryness. After cooling, add 5 ml of hydrochloric acid(60) (1:1) and a little amount of water, and dissolve it by heating. After adding a few drops of Methyl Orange solution(47), neutraluze with ammonia solution(61) (1:2), and precipitate iron or aluminum component by adding two or three drops of ammonia water. Filtrate the precipitate through a filter paper(62) and wash it sufficiently with warm water. Neutralize the filtrate and washings (about 150 ml) with hydrochloric acid (1:1) and, adding superfluously 2 ml of the acid, heat it to nearly boiling. Add 10 ml of hot barium chloride solution(63) (10 %) by sufficient agitation to mix, heat for one hour on a water bath, and then let it stand for one day and night. Filtrate this through a filter paper, and wash with warm water until no chloride ion(64) is found. Place the precipitate in a crucible together with a filter paper, and heat carefully to incinerate the filte paper. After cooling, moisten with one drop of sulfuric acid(32), and heat gently, which is followed by strong heating for 30 minutes at about 800°C. After cooling in a desiccator, weigh the mass. Separately carry out a blank test similarly to the procedure to correct the error caused by impurity of reagents. Calculate sulfur content according to the following formula, and express the average of two tests (to the third decimal place).

$$\text{Sulfur content (\%)} = \frac{(A-B)\times 0.1373}{D}\times 100$$

where A : mass after igniting and cooling (g)

B : mass of blank test (g)

D : absolute dry mass of sample (g)

Notes (57) Dissolve 320 g of the guaranteed grade reagent specified in JIS K 8567 in water up to 1 l. Concentration is 50 g/l with the conversion to MgO.

(58) Use the guaranteed grade reagent specified in JIS K 8541.

(59) Use the guaranteed grade reagent specified in JIS K 8223.

(60) Use the guaranteed grade reagent specified in JIS K 8180.

(61) Use the guaranteed grade reagent specified in JIS K 8085

(62) Use class 5 C specified in JIS P 3801.

(63) Use the guaranteed grade reagent specified in JIS K 8155

(64) Confirm chloride ion by silver nitrate solution (5 %) that is prepared by dissolving 5 g of the guaranteed grade reagent specified in JIS K 8550 in water up to 100 ml.

Remarks 1. In the case of dull condition of the sample, make the quantity of magnesium nitrate solution 5 ml, and carry out another operation of filtration after dissolving in hydrochloric acid and water by heating.

2. This is applied to rayon.

3. The apparatus used for the tests shall be in accordance with JIS K 0050.

7.29 <u>Mean Polymerization Degree</u> Weigh accurately the sample corresponding to 0.01 g as absolute dry mass, and put it in the viscometer shown in Fig. 13 (0.8 to 1.0 mm in capillary diameter, 12 cm in length, and dropping time of 20 to 40 seconds for 3 ml of copper oxide ammonia solution). After replacing completely the air inside with purified hydrogen or nitrogen gas, add 10 ml of copper oxide ammonia solution (11 g of copper, 210 g of ammonia solution, and 10 g of cane suger in 1 l) at 20°C under flowing nitrogen or hydrogen to intercept atmosphere and, after 5 minutes, standing still, shake it about 10 minutes (15 cm amplitude and 100 times shuttling) in order to dissolve completely. Allow the solution stand still in a thermostatic bath at 20 ± 0.05°C, and after 1 minute shaking and 30 minutes later from dissolving operation, measure the time for dropping. Express the viscosity by relative viscosity based on that of copper oxide ammonia solution(65)(66), and express the average of two test results (to the third decimal place).

Calculate the mean polymerization degree according to the following formula (by integer):

Specific viscosity of cellulose $\eta_{SP}=\eta_{rel}-1$

Mean polymerization degree $= \dfrac{\eta_{SP}}{C \cdot K_m}$

where η_{SP} : specific viscosity of cellulose

η_{rel} : relative viscosity

C : number of gram of sample in 1 l solution

K_m : viscometric molecular weight constant 5×10^{-4}

Notes (65) Preparation of Coper Oxide Ammonia Solution Place pure copper scraps, 2 *l* of ammonia solution (28 %) cooled sufficiently, and 20 g of cane sugar in the apparatus for preparation of copper oxide ammonia solution, and send air inside by sucking it. Four to five hours is necessary to prepare the solution.

In summer, it is preferable to cool the apparatus shut off from outside. After operation, transfer it in a reagent bottle covered with brown or black paper, and keep it by tightly stoppering in a dark place. Copper sulfate is available for this preparation.

(66) Testing Method for Copper Oxide Ammonia Solution

(1) Determination of Copper Take 5 ml of copper oxide ammonia solution and, after adding 55 ml of purified water, 15 ml of buffer solution, 15 ml of methanol, and a few drops of PAN indicator, titrate with 1/30 M EDTA standard solution. Make the point when blue violet turns to yellow green as the end point, and read the volume required for titration. Calculate the quantity of copper contained in 1 *l* of cooper oxide ammonia solution according to the following formula.

$$\text{Copper } (g/l) = \begin{matrix}\text{volume of 1/30 M} \\ \text{EDTA solution (ml)}\end{matrix} \times \text{factor} \times 200$$

(1.1) 1/30 M EDTA Standard Solution Dissolve 12.6 g of the guaranteed grade reagent disodium ethylenediaminetetraacetic acid specified in JIS K 8107 in 1 *l* of purified water. Separately, take accurately 25 ml of 1/30 M copper standard solution [Dissolve 2.118 g of copper (99.999 %) in 10 ml of concentrated nitric acid, and dilute it with purified water up to 1 *l*.], and add 5 ml of concentrated aqueous ammonia, 15 ml of buffer solution, 25 ml of methanol, and a few drops of PAn indicator solution. Titrate this solution with above EDTA solution, and read the volume required for titration by the end point that blue violet turns to yellow green. Calculate a factor of EDTA solution according to the following formula:

$$\text{Factor} = \frac{25}{\begin{matrix}\text{Volume of 1/30 M EDTA} \\ \text{solution for titration}\end{matrix}} \times 0.00218$$

(1.2) Pan Indicator This is 0.1 % methyl alcohol solution of 1-pyridylazo-2-naphthol.

(1.3) Buffer Solution Dissolve 90.6 g of the guaranteed grade reagent sodium acetate specified in JIS K 8372 in 600 ml of purified water, and add further 400 ml of the guaranteed grade reagent acetic anhydride specified in JIS K 8886 to prepare.

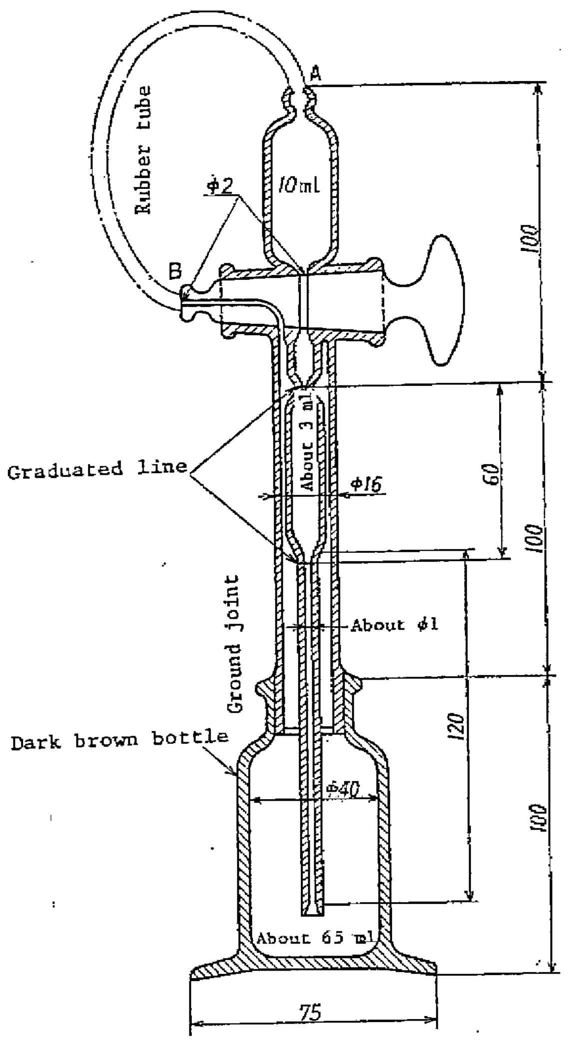
(2) Determination of Ammonia Take 1 ml of the sample in about 50 ml of cold water, and titrate it with 1 N sulfuric acid or hydrochloric acid by dropping methyl orange solution[47] as indicator. 12.4 ml for titration is sutiable for per 1 ml of the sample.

Remark 1. This is applied to rayon and cupro.

2. The appliances used for test shall be in accordance with JIS K 0050.

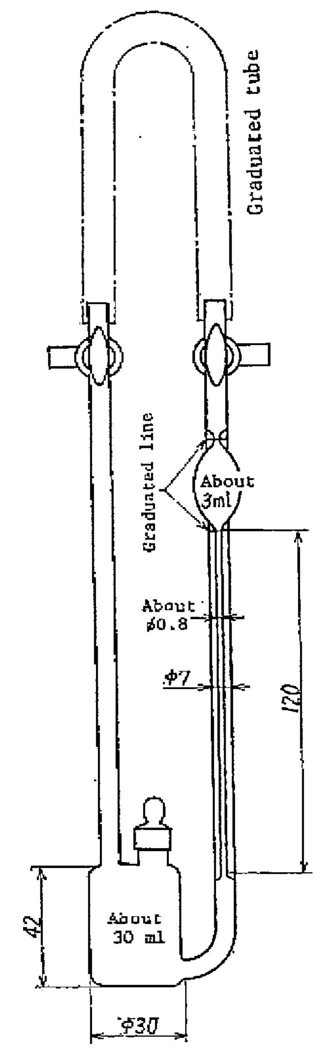
Fig. 13. Viscometer

Oken-Type Viscometer
Unit: mm



Glass ball contained in bottle
(diameter 6 mm) 25 pieces

Nakano-Type Viscometer
Unit: mm



Glass ball
(diameter 6 mm) 10 pieces

7.30 Degree of Exhaustion After combing sufficiently the sample under moisture equilibrium with a hand card, weigh out 3 g of the sample in water at 70°C of 100 times the sample quantity, and wash it by stirring to mix for 30 minutes. After dehydrating, add water so as to make the mass of wet sample 5 times the original mass, and dry the sample under following conditions:

Dyestuff(67)	Direct skyblue 6 B (C 1 direct blue-1) Weigh 0.2 % of the object to be dyed.
Auxiliaries	Sodium sulfate anhydride, 20 % of the object to be dyed
Bath ratio	100 : 1
Temperature	45 ± 1°C
Stirring to mix	Once per 20 sec.

Let the residual dying liquid cool to room temperature, and by means of photoelectric colorimeter(68) measure absorbance. Obtain the degree of exhaustion from the working curve(69) prepared separately, and express the average of two testing results (to the first decimal place).

Notes (67) Purified dyestuff is used after conversion to an anhydride.

(68) A photoelectric colorimeter is under the following condition.

Filter 620 to 630 nm

(69) Dilute the dyeing solution prepared in 7.30 with sodium sulfate solution (2 g/l) by following ratio, and measure the absorbance respectively.

Table 6

Degree of exhaustion %	Aliquot of dyeing solution ml	Volume of 2 g/l sodium sulfate solution ml
100	0	100
80	20	80
60	40	60
40	60	40
20	80	20
0	100	0

Remark: This is applied to rayon.

7.31 Degree of Acetylation

Method A (Direct Method) Weigh out about 5 g of the sample and, after removing oily component according to the method of (3) Extract by Diethyl Ether(70) in 7.22 Solvent Extractable Matter, as appropriate, cut it to about 5 mm length after drying. Take about 1 g of the sample in a 20 mm diameter weighing bottle and, after drying in a thermostatic dryer at 105 ± 2°C until it attains constant weight, cool in a tightly closed desiccator. After weighing accurately the sample as it is in the weighing bottole, transfer it in a 300 ml conical flask with a stopper, and obtain the absolute dry mass of the sample after weighing accurately the mass of the weighing bottle. Then, add 40 ml of 75 % ethanol(37) in the flask, and heat it for 30 minutes at 55 ± 5° after slightly stoppering it. Add 40 ml of 0.5 N sodium hydroxide solution(42), and, after heating for 15 minutes at 50 ± 5°C, let it stand for 48 hours at room temperature. After adding several drops of phenolphthalein solution(44), back-titrate with 0.5 N hydrochloric acid(60). when disapearing pink color, add further 2 ml of 0.5 N hydrochloric acid, and let it stand for 12 hours. Next, titrate with 0.5 N sodium hydroxide solution. Separately, carry out a blank test completely similarly, and calculate the degree of acetylation (%) according to the following formula. Carry out twice the test, and record the average of them (to the first decimal place).

$$\text{Degree of acetylation (\%)} = \frac{[(A-B)\times F_b-(C-D)\times F_a]}{W}\times 3.0025$$

where A : required volume of 0.5 N sodium hydroxide solution (ml)

B : required volume of 0.5 N sodium hydroxide solution for blank test (ml)

F_b : factor of sodium hydroxide solution

C : required volume of 0.5 N hydrochloric acid (ml)

D : required volume of 0.5 N hydrochloric acid for blank test (ml)

F_a : factor of hydrochloric acid

W : absolute dry mass of sample (g)

Note (70) Instead of diethyl ether extracting method, other solvent extracting methods may be used. In this case, append a note stating the method adopted.

Method B (Dissolving Method) Similarly to Method A, weigh accurately about 0.5 g of the sampe in a flask. Add 50 ml of purified acetone[71] in the flask to dissolve completely, and after adding 50 ml of 0.2 N sodium hydroxide solution[42], let it stand for 3 hours at 25 ± 2°C by stirring sometimes. Then add 50 ml of 0.2 N hydrochloric acid[50] and stir it sometimes. Fifteen minutes later, by adding phenolphthalein solution[44] as indicator, titrate with 0.2 N sodium hydroxide solution[42]. Separately, carry out a blank test completely and similarly, and calculate the degree of acetylation (%) according to the following forumla. Carry out twice the test, and express the average of them (to the first decimal place).

$$\text{Degree of acetylation (\%)} = \frac{(A-B)\times F\times 1.201}{W}$$

where A: required volume of 0.2 N sodium hydroxide (ml)

B: required volume of 0.2 N sodium hydroxide for blank test (ml)

F: factor of 0.2 N sodium hydroxide solution

W: absolute dry mass of sample (g)

Note (71) Use the guaranteed grade reagent specified in JIS K 8034.

Method C (Dissolving Method) Similarly to Method A, weigh out accurately about 2.0 g of the sample in a flask. Add 70 ml of purified acetone[71] in the flask, and let it stand for 10 minutes. Dissolve the sample completely by adding 30 ml of dimethyl sulfoxide.

Then, stir it for 5 minutes by adding 50 ml of purified acetone and, after adding 30 ml of 1 N sodium hydroxide solution[42], stir it for two hours at 25 ± 2°C. After saponification, add 100 ml of warm water at 50°C, and stir it for 15 minutes. By adding several drops of phenolphthalein solution[44], titrate it with 1 N sulfuric acid[32] until it becomes colorless. Separately, carry out a blank test completely and similarly, and calculate the degree of acetylation (%) according to the following formula. Carry out twice the test, and express the average of them (to the first decimal place).

$$\text{Degree of acetylation (\%)} = \frac{(B-A)\times F\times 6.005}{W}$$

where A: required volume of 1 N sulfuric acid (ml)

B: required volume of 1 N sulfuric acid for blank test (ml)

F: factor of 1 N sulfuric acid

W: absolute dry mass of sample (g)

Remarks 1. Method A is to every acetate, Method B to the acetate except triacetate, and Method C to triacetate. Append a note stating the method adopted.

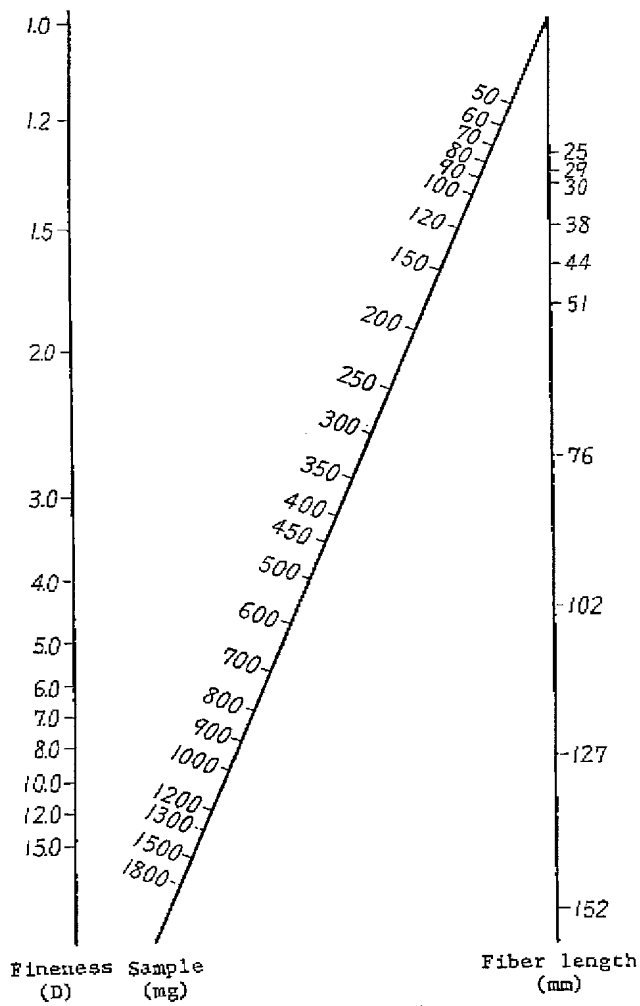
2. When titanium oxide, pigment, or others are added, subtract the mass of these additives from the absolute dry mass.

3. The appliances used for the tests shall be in accordance with JIS K 0050.

Attached Table Density of Fibers

Kind of fibers	Density g/cm^3
rayon	1.50 to 1.52
(polynosic)	1.50 to 1.52
cupro	1.50 to 1.52
acetate	1.32
(triacetate)	1.30
nylon	1.14
vinylon	1.26 to 1.30
vinylidene	1.70
polyvinyl chloride	1.39
polyester	1.38
acrylic	1.14 to 1.17
modacrylic	1.28
polypropylene	0.91
polychlal	1.32

Attached Figure. Nomograph for Fiber Length Diagram and Specimen (Nomograph based on 120 mg of specimen weight when fineness is 1.5 denier and fiber length is 38 mm in the case of rayon.)



Reference

1. Calculation Methods

1.1 Percentage of Deviation Percentage of deviation shall be calculated according to the following formula:

$$\text{Percentage of deviation (\%)} = \frac{a-b}{b} \times 100$$

where a : average of measured values

b : indicated value

1.2 Coefficient of Variation Coefficient of variation shall be calculated according to the following formula:

$$\text{Coefficient of variation (\%)} = \frac{\hat{\sigma}}{\bar{x}} \times 100$$

where $\hat{\sigma} = \sqrt{\sum (x-\bar{x})^2/(n-1)}$

n : number of all measurings

x : each measured value

$\bar{x}$: average of all values

Remark: The value of $\hat{\sigma}$ may be obtained according to the following formula:

$$\hat{\sigma} = \frac{\bar{R}}{d_2}$$

where $\bar{R}$: average of R

R : difference between the largest and smallest of measured values

d_2 : estimation factor of population standard deviation

The value of d_2 is shown in Reference Table 1. In cases where the size of the sample is larger than 10, obtain $\bar{R}$ by dividing at random the sample into equally-sized small groups.

Reference Table 1

n'	d_2	n'	d_2	n'	d_2
2	1.128	7	2.704	12	3.258
3	1.693	8	2.847	13	3.336
4	2.059	9	2.970	14	3.407
5	2.326	10	3.078	15	3.472
6	2.534	11	3.173		

Remark: n' in the above table means the size of the sample in a group of measured values.

1.3 Strength Ratio at Wet and Dry Strength ratio at wet and dry shall be calculated according to the following formula:

$$\text{Strength ratio at wet and dry (\%)} = \frac{S_W}{S_D} \times 100$$

where S_D : tensile strength at standard state (gf) {N}

S_W : tensile strength at wet state (gf) {N}

1.4 Knot Strength Ratio Knot strength ratio shall be calculated according to the following formula:

$$\text{Knot strength ratio } (\%) = \frac{S_{KD}}{S_D} \times 100$$

where S_D : tensile strength at standard state (gf){N}

S_{KD} : knot strength at standard state (gf){N}

1.5 Loop Strength Ratio Loop strength ratio shall be calculated according to the following formula:

$$\text{Loop strength ratio } (\%) = \frac{S_{LD}}{S_D} \times 100$$

where S_D : tensile strength at standard state (gf) {N}

S_{LD} : loop strength at standard state (gf) {N}

2. Way to Find Number of Tests

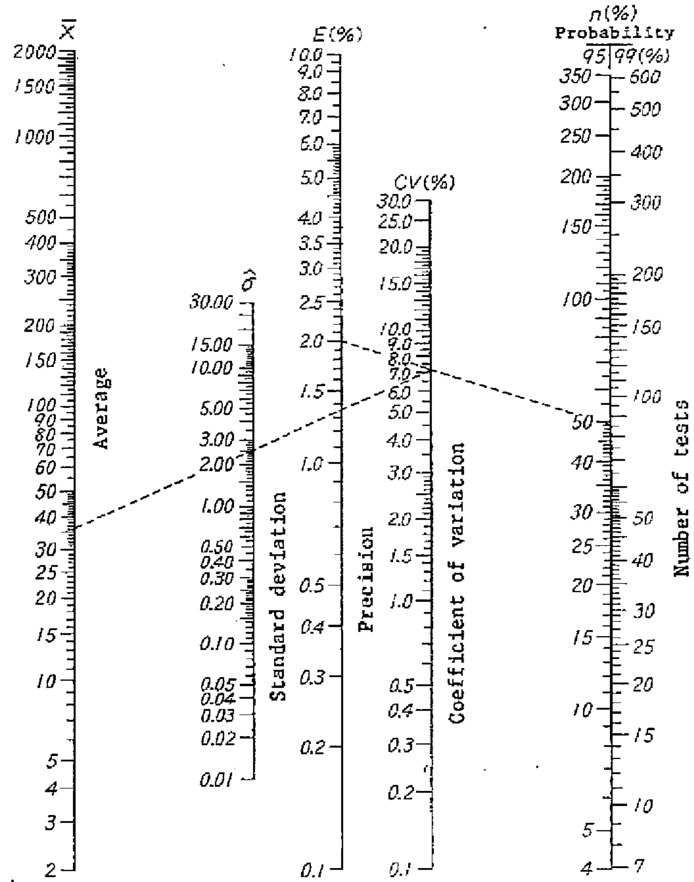
When the number of tests is to be decided between the parties concerned, the following method is suitable.

The number of tests is obtained by using Reference Fig. 1 Nomograph for Number of Tests and Reference Fig. 2 Nomograph for Number of Tests in accordance with the coefficient of variation (%) (the percent value obtained by dividing estimate value(1) of standard deviation by the average value), and the precision(2) and probability which are required. The nomograph for number of tests is used as follows; draw a straight line connecting coefficient of variation and precision, find the intersecting point between this line and a test number line, and read the number of tests according to the probability. Raise to a unit the fraction thereof.

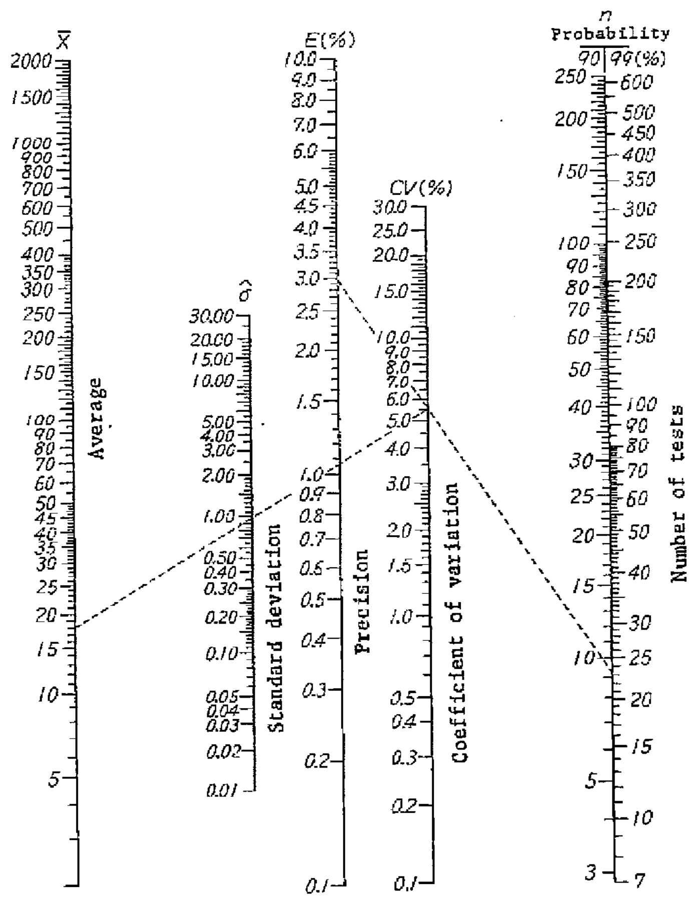
Note (1) The method to obtain the estimate value of standard deviation shall be according to 5.1 of The Explanation of Test and Estimation on Population Mean in JIS Z 9042 to Z 9053, as appropriate.

(2) Precision means the percent value obtained by dividing the difference of the averages referred to by the average.

Reference Fig. 1. Nomograph for Number of Tests



Reference Fig. 2. Nomograph for Number of Tests



Applicable Standards:

JIS K 0050-General Rules for Chemical Analysis

JIS K 0061-Testing Methods for Specific Gravity of Chemical products

JIS K 8034-Acetone

JIS K 8085-Ammonia Water

JIS K 8102-Ethanol (95)

JIS K 8103-Diethyl Ether

JIS K 8107-Ethylenediaminetetraacetic Acid Disodium Salt

JIS K 8155-Barium Chloride

JIS K 8180-Hydrochloric Acid

JIS K 8223-Perchloric Acid

JIS K 8230-Hydrogen Peroxide

JIS K 8271-Xylene

JIS K 8355-Acetic Acid

JIS K 8372-Sodium Acetate, Anhydrous

JIS K 8459-Carbon Tetrachloride

JIS K 8541-Nitric Acid

JIS K 8550-Silver Nitrate

JIS K 8567-Magnesium Nitrate

JIS K 8576-Sodium Hydroxide

JIS K 8680-Toluene

JIS K 8799-Phenolphthalein

JIS K 8858-Benzene

JIS K 8886-Acetic Anhydride

JIS K 8891-Methanol

JIS K 8893-Methyl Orange

JIS K 8937-Ligroin

JIS K 8951-Sulfuric Acid

JIS K 8960-Ammonium sulfate

JIS L 0101-Tex System to Designate Linear Density of Fibres, Yarn Intermediates, Yarns and Other Textile Materials

JIS P 3801-Filter Paper (for Chemical Analysis)

JIS R 3503-Glass Apparatus for Chemical Analysis

JIS Z 8401-Rules for Rounding off of Numerical values

JIS Z 8701-Specification of Colours According to the CIE 1931 Standard Colorimetric System and the CIE 1964 Supplementary Standard Colorimetric System

JIS Z 8703-Standard Atmospheric Conditions for Testing

JIS Z 8722-Methods of Measurement for Colour of Reflecting or Transmitting Objects

JIS Z 8806-Methods of Humidity Measurement

Reference Standards:

JIS Z 8203-SI Units and the Use of their Multiples and of Certain other Units

JIS Z 9042-Significance Test of Difference Between the Population Mean and the Standard (Standard Deviation Known, One-Sided)

JIS Z 9043-Significance Test of Difference Between the Population Mean and the Standard (Standard Deviation Known, Two-Sided)

JIS Z 9044-Significance Test of Difference Between the Population Mean and the Standard (Standard Deviation Unknown, One-Sided)

JIS Z 9045-Significance Test of Difference Between the Population Mean and the Standard (Standard Deviation Unknown, Two-Sided)

JIS Z 9046-Significance Test of Difference Between the Two Population Means (Standard Deviations Known, One-Sided)

JIS Z 9047-Significance Test of Difference Between the Two Population Means (Standard Deviations Known, Two-Sided)

JIS Z 9048-Significance Test of Difference Between the Two Population Means (Standard Deviations Unknown, One-Sided)

JIS Z 9049-Significance Test of Difference Between the Two Population Means (Standard Deviations Unknown, Two-Sided)

JIS Z 9050-Interval Estimation of the Population Mean (Standard Deviation Known)

JIS Z 9051-Interval Estimation of the Population mean (Standard Deviation Unknown)

JIS Z 9052-Interval Estimation of the Difference between Two Population Means (Standard Deviations Known)

JIS Z 9053-Interval Estimation of the Difference between Two Population Means (Standard Deviations Unknown)